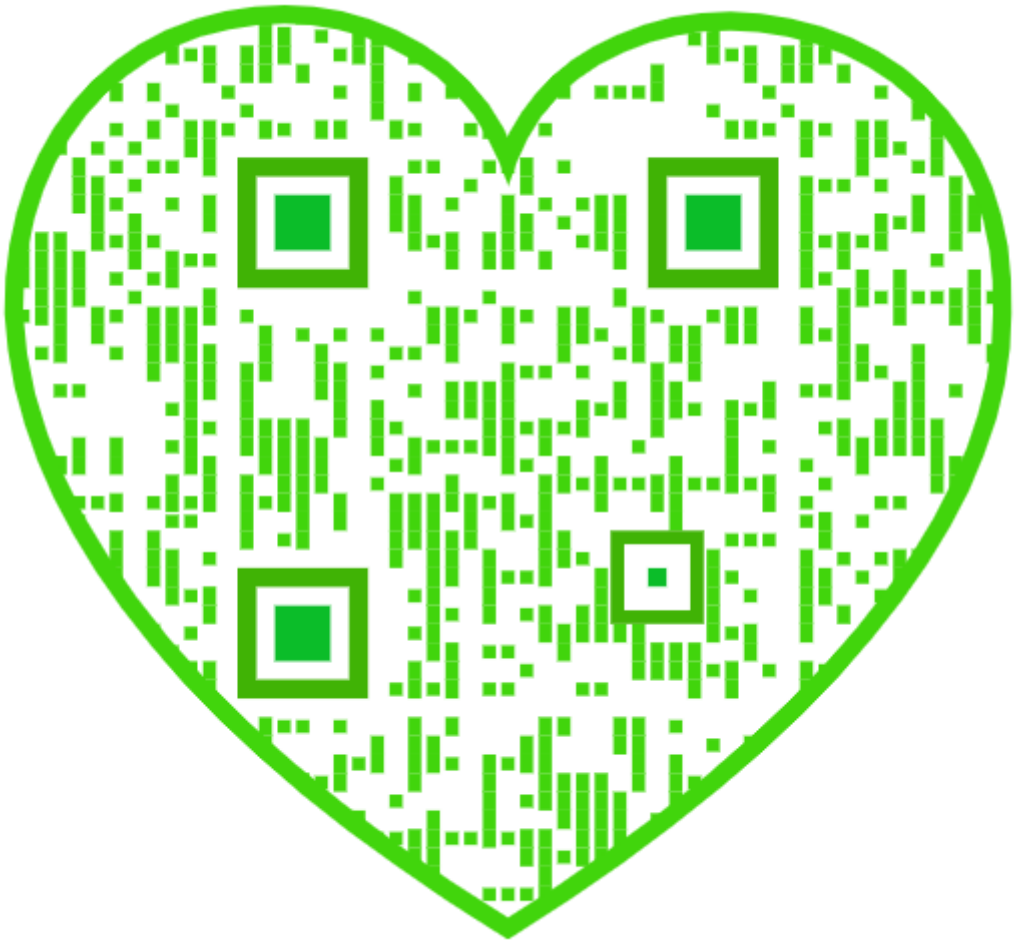

LABORATORY MANUAL

OF

CHEMISTRY

FOR SECONDARY SCHOOLS

BY

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NEW EDITION

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PREFACE

In the preparation of this manual, it has been the aim of the authors to present a series of experiments for students beginning chemistry. It is believed that those experiments involving exact measurements are judiciously arranged and not too difficult for high school use. Special care has been taken in the selection in order that the apparatus required may be of simple character and in most cases inexpensive.

Having had many years' experience in secondary schools, the authors feel justified in asserting that most text-books on chemistry have too many experiments, so elementary in character that they leave no lasting impression on the minds of the pupils, thereby weakening the desire for scientific investigation instead of strengthening it. Fewer experiments, requiring more careful thought and manipulation of apparatus produce much better results.

While some of the material has been gathered from various sources, the treatment is independent. Especially do the authors wish to recognize the assistance given by some of our modern text-books, and for the kind hints and suggestions of Dr. George A. Hulett, Mr. N. H. Williams, and Prof. George O. Higley.

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LABORATORY MANUAL OF CHEMISTRY

THE BALANCE

In order to weigh any substance accurately, or to detect minute differences in the weights of different things, it is necessary to employ a delicate balance and exact weights. For instance, a few grains of sand sprinkled upon the scale of an ordinary kitchen balance will make absolutely no dif-

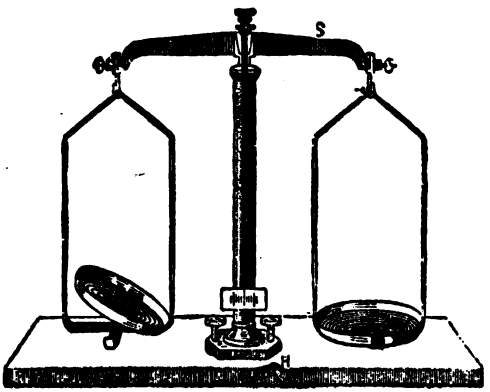

Fig. 1.

ference, whereas a single particle of sand placed upon one pan of a chemical balance would completely weigh it down. The chemical balance is usually enclosed in a glass case, partly to protect it from dust and dirt, and partly in order

Note.—When not in use, the balance and the weights should not be left exposed to the laboratory atmosphere, but should be either covered with a glass shade, or put away into a cupboard.

that it may not be exposed to the slightest draft when being used. Such a balance will readily weigh to a fraction of a milligram.

For the purposes of the elementary student, an instrument of the extremest delicacy is not necessary or desirable. Fig. 1 shows a balance suitable for his requirements.

By turning the handle or lever H, the beam is liberated from its support, and is then free to swing. This balance will turn with 2 milligrams, and is able to carry as much as 100 grams. It must not be used for heavier weights than this.

The set of weights consists of the following: 50, 20, 10, 10, 5, 2, 1, 1, 1, grams, making up 100 grams; and 0.5, 0.2, 0.1, 0.1, 0.05, 0.02, 0.01, 0.01.

Volumes and Weights

Experiment 1: Find the weight of several different volumes of water.

a. Weigh the beaker.

b. Fill the burette with water nearly to the top of the graduated portion. Note and record the exact reading in c. c. and 1/10 c. c. Next, by opening the valve at the bottom of the burette, let water run into the beaker. Close the valve when from 10 to 15 c. c. has passed into the beaker. Note and record again the exact reading. Query: How do you find the volume?

c. Weigh the beaker with water in it and record.

d. Using the burette as before, place about 30 c. c. in the beaker, weigh and record. Repeat, using successively 40, 50, and 100 c. c.

For the note-book:

1. Name the experiment.
2. Name materials and apparatus.

3. Operations and observations.

4. Tabular form:

No. of trial.	First reading (burette).	Second reading.	Volume.	Weight of beaker empty.	Weight of beaker and water.	Weight of water.	Ratio weight of 1 c.c. water.
1	c. c.	c. c.	c. c.	g.	g.	g.	
2							
3							
4							

5. Conclusion: Ratio of weight to volume in weight is expressed in grams and volume in c. c. of water.

MATTER

Definition (*Write in note-book*).

Three forms of matter:

(a) Solid.

(b) Liquid.

(c) Gaseous.

Matter is made up of small particles called *molecules*.

Molecules are composed of *atoms*.

NOTE.—Most chemists suppose the molecule to be the smallest particle of matter that exists alone, while the atom is the smallest particle that can enter into combination. In a few cases of elements the atom and the molecule are identical.

(a) A *solid* is a substance whose molecules change their position with great difficulty; e. g., iron, ice.

(b) A *liquid* is a substance whose molecules glide freely about each other; e. g., water, mercury.

(c) A *gas* is a substance whose molecules tend to separate and get farther away from each other; e. g., hydrogen, oxygen.

CHEMISTRY

Definition (*Write in note-book*).

History of, three periods:

1. Alchemy.
2. Phlogiston.
3. Scientific.

Force: That which produces or tends to produce a

Kinds of change:

(a) Wind blowing.
(b) Horse drawing a wagon.
(c) Heat.
(d) Light.
(e) Acid acting upon a metal.
(f) Gravitation.

Kinds of change:

(a) Physical.
(b) Chemical.

Physical change is one in which the identity of the body is not destroyed.

Chemical change is one in which the identity of the body is destroyed.

Forces that may produce a physical change:

(a) Heat.
(b) Steam.
(c) Animal force, etc.

Force that produces chemical change: A peculiar force called *chemical affinity.*

Physical force may act through very long distances, such as may easily be seen or measured.

Chemical force acts through insensibly short distances, such as cannot be measured or seen, even with the most powerful microscope.

Agents sometimes required to produce chemical change:

(a) Heat.
(b) Cold.
(c) Light.
(d) Heavy blow.
(e) Light blow.
(f) Moisture.
(g) Absence of moisture.

Substance may be *simple* or *compound.* By *simple* is meant one from which no other kind can be obtained; e. g., gold, lead, copper, hydrogen, oxygen, mercury. Such a simple substance is called an *element.*

A *compound* substance is one composed of two or more simple substances and from which these substances may be obtained; e. g., common salt, chemically known as sodium chloride, whose chemical formula is written NaCl, the Na representing the simple element sodium (natrium) and the Cl representing the simple element chlorine.

In chemistry, to represent the different elements, a kind of shorthand is used which is the first letter of the Latin name of the element; or, if two or more begin with the same letter, then a distinguishing letter is written with the first letter.

There are about 75 elements thus far discovered.

NOTE.—See table of elements in text.

Bunsen Burner

Experiment 2: Note the effect of opening and closing the holes at the base of the burner. Which gives the luminous flame? Hold a piece of crayon in the upper part of the luminous flame. Note the black deposit. What is it? Now open the holes at the base and rub together two pieces of charcoal, holding them as near the open

holes as possible. Do the two experiments suggest the cause of luminosity in the flame? What is it?

Experiment 3: Hold a match across the flame of a Bunsen burner near the top of the burner. When the match begins to burn, remove it, extinguish the flame, and note where the match is charred. Explain. Stick a pin through a sulphur match about one centimeter from the end, suspend in the top of the burner, and then light the gas. Explain the result. Find the hottest part by holding a platinum or copper wire at different parts of the flame when a full current of gas is burning.

Experiment 4: With the holes open, carefully turn the gas very low until the flame "strikes back." Explain. Now turn on the full current of gas and strike the rubber hose a sharp blow. This will often bring the flame to the top of the burner. The gas must not be permitted to continue burning at the base.

FIG. 2.

Summary.—What is the object of the holes?

Why does the gas burn at the top, and not inside?

Why does the flame sometimes "strike back" when the gas is turned low?

Why is the flame non-luminous with the holes open?

Physical and Chemical Changes

Experiment 5: Dissolve as much sugar as possible in half a test-tube of cold water. Kind of change? Next, holding the test-tube in an inclined position, pour in sulphuric acid carefully until a layer of about 1 cm. in depth forms in the bottom of the tube below the sugar solution. What change in color appears in the sugar solution near the sulphuric acid? Next pour off most of the unchanged sugar solution, and note that the charred sugar does not return to its original condition; the change is permanent. Is it a physical or a chemical change? What change in temperature occurred upon adding the sulphuric acid to the solution?

Experiment 6: Heat a platinum wire in the flame. Remove from flame. Kind of change? With the tongs hold a piece of magnesium ribbon in the flame. Result? After the product cools does it return to the original condition as the platinum wire does on cooling? What kind of change is represented?

Experiment 7: Heat one gram of mercuric oxide in a test-tube until the color changes, then pour out on a piece of paper and allow to cool. What is the result? What kind of change? Pour the mercuric oxide back into the test-tube and heat strongly for several minutes, until globules of a silvery substance collect in the cooler part of the tube. Upon standing until thoroughly cool, does this substance return to the original condition? What kind of change is here represented?

Chemical Change

Experiment 8: Take a piece of copper the size of a small nail-head, and, having placed it in the evaporating dish, pour upon it 4 c. c. of nitric acid (HNO_3) and place

it under the hood, carefully noticing all that takes place until the action ceases. Now pour in about 10 c. c. of water and evaporate to dryness. Has the copper changed its appearance? Can you bring it back to its metallic state? What kind of change? Again, pour water upon the dry salt and place within the solution a bright iron nail. What takes place?

Mixture Changed to Compound by Heat

Experiment 9: On the side table find some of the substance provided for this experiment. Examine carefully and determine the constituents if possible. Next pass a magnet several times through the mixture and determine whether the constituents can be separated in this way. Is this means classed as physical or chemical? What is the mixture?

Experiment 10: Place two or three grams of the substance in a test-tube and heat strongly. After heating to a low redness, remove the test-tube from the flame and see whether the glow spreads through the mass. Of what is continued evolution of heat characteristic? As soon as the action has ceased, break the test-tube and compare the resulting substance in color, texture, etc., with the original. What kind of change is illustrated? To what class of substances does the product belong? What is the chemical name of the substance?

For the note-book:

1. Name of experiment.
2. Operations and observations.
3. Discussion. Reasoning leading up to the conclusion.
4. Conclusion. Distinction between mixture and compound; form of action used in producing the change.

Separation of Constituents of a Mixture

Experiment 11: Place about ½ a gram of gunpowder in a test-tube, add 2 c. c. of carbon bisulphide and allow it to stand for ten minutes. Pour out the solution upon a watch-glass and let stand until the liquid has evaporated. What is the yellow substance deposited on the glass? From what was it obtained? What is one of the constituents of gunpowder?

Experiment 12: Next place the gunpowder, which has already been treated with carbon bisulphide, in a test-tube with 5 c. c. of water and warm for a few minutes. Pour out the solution into an evaporating-dish, evaporate carefully to dryness, and compare the resulting crystalline substance with known potassium nitrate. Inference? Finally, examine the remains of the gunpowder which has been subjected to the two solvents, carbon bisulphide and water, and see if you can determine what is the third constituent of gunpowder.

Summary.—Have you used physical or chemical means for separating the gunpowder into its constituents? Is the gunpowder a mixture or a compound? After burning gunpowder, can the same means be employed for separating the constituents? What are the products?

HYDROGEN

Action of Sodium on Water

Experiment 13: *a.* Fill a test-tube with water, and having placed your thumb over the mouth, invert it over the pneumatic trough, as shown in figure.

b. Now take a perfectly clean piece of sodium the size of a pea, and place it in a small piece of lead pipe, or wire gauze. After having closed the pipe by pressing the sides

together, quickly insert the same under the mouth of your t. t. in the p. t. Note what takes place.

c. After the t. t. is full of gas, raise the t. t., mouth downward, from the water and quickly apply a lighted match. What follows?

What had you in your t. t.? Was it air? Why? This gas is called *hydrogen,* and is an element. Whence came it?

Experiment 14: Take a perfectly clean evaporating-dish and place therein 10 c. c. of water. Now take a piece

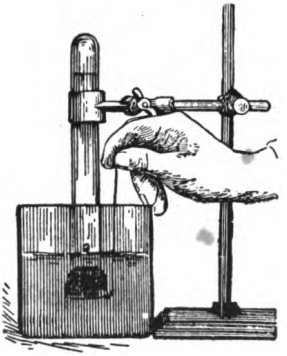

FIG. 3.

of red litmus paper and dip it into the water; does the paper change in color? Now throw upon the water in the evaporating-dish a tiny piece of sodium, and after the reaction has ceased, again dip into the water the piece of red litmus paper. Now what happens? What has become of the sodium?

The sodium split off, as it were, the gas (hydrogen) from the water and took its place and formed another compound called sodium hydroxide.

Preparation of Hydrogen

Experiment 15: Take from your work-table 5 g. of zinc, and having placed it in a generating flask, from which passes a delivery tube as in Fig. 4, pour over the zinc a mixture of 10 c. c. of sulphuric acid and 50 c. c. of water previously mixed and allowed to cool. CAUTION:

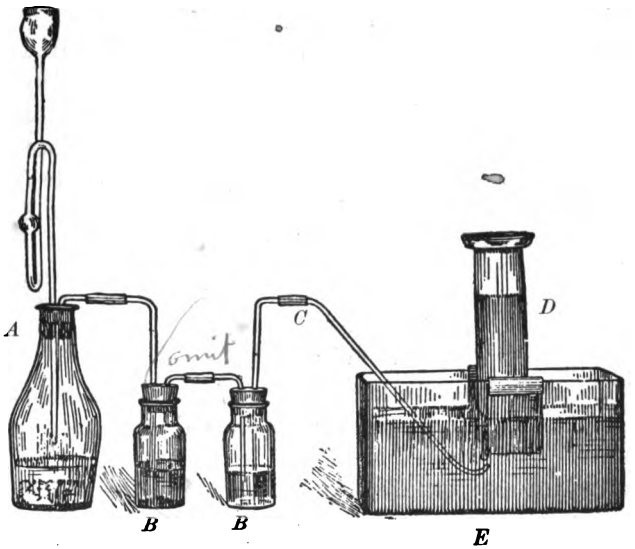

FIG. 4.—A, generating flask. B B, wash bottles. C, delivery tubes. D, cylinder for collecting gas. E, pneumatic trough.

Text, p. 9*.

In mixing an acid with water, pour the acid into the water, and not the water into the acid. Now pass the mouth of your delivery tube under the test-tubes, previously filled over the pneumatic trough, and collect the gas

* Hessler and Smith is the text referred to in all instances in this manual.

given off, as in the case of the decomposition of water. What do you think the gas is? Test it. Has it any color? Where did the gas come from? Where has the zinc gone? Pour the liquid from your generating bottle into an evaporating-dish, place on your retort stand and sand bath and slowly evaporate to dryness. What have you? This is called a salt. There are other ways of forming salts that will be noticed hereafter.

The gas obtained from the last experiment behaved in exactly the same way as that obtained by the action of sodium upon water. In fact, it is the same gas; it came from the sulphuric acid, while the zinc took its place in the acid, so that, instead of having hydrogen sulphate (or, as it is usually called, sulphuric acid), we now have zinc sulphate, which has the following formula: $ZnSO_4$. The reaction would be expressed upon paper by a chemical equation thus:

$$Zn + H_2SO_4 = ZnSO_4 + 2H$$

The symbols on the left are called *factors*, and those on the right are called *products*. Notice that it takes one atom of zinc to take the place of two atoms of H. This is often the case, one element having double the replacing power of another. You see a chemical equation expresses the elements taken, the parts by weight and also by volume. (Teacher gives one or two additional experiments under hydrogen.)

Additional Tests with Hydrogen

Experiment 16: CAUTION: *a.* Collect three bottles full of hydrogen. Holding one of the bottles downward, insert a blazing splinter. Result? Is the gas combustible? Does it support combustion?

b. Holding a wider-mouthed jar in the left hand, pour

the gas from the second bottle *upward* into the jar. How can you show that the gas has passed into the jar? What does this show about the weight of hydrogen compared with air? Burn the gas in the third bottle.

Hydrogen mixed with air is violently explosive if brought to a flame. If in an open test-tube, however, the explosion is harmless, as the flame simply shoots from the mouth of the tube and the glass is not broken. Only when the gases are confined is the explosion dangerous. To safely illustrate this property, proceed as follows:

c. Fill a test-tube half full of water, invert in the pneumatic trough, and pass in hydrogen until the water is all displaced from the tube. The test-tube now contains half hydrogen and half air. Place the thumb over the mouth of the test-tube and bring near a flame. The sharp whistle produced upon burning is proof of hydrogen mixed with air. The experiment may be varied by filling the test-tube *full* of water and displacing with hydrogen. The pure hydrogen burns quietly.

For the note-book. Outline of the hydrogen experiment.

I. Apparatus.
II. Materials.
III. Diagram.
IV. Method of making:
    1. Action.
    2. Collection of hydrogen.
    3. Salt in solution.
V. Equation for action.
VI. Tests:
    1. ?
    2. ?
    3. ?
    4. Class experiment in diffusion.

VII. Properties of hydrogen.

1. Physical: State; color; odor; density; solubility; diffusibility.
2. Chemical: Non-supporter of combustion; combustible; explosive when mixed with air.

Kipp's apparatus, same method.

VIII. Other methods of making hydrogen.

1st method: Kipp's apparatus, same method.
2d " Sodium on water.
3d " Iron on steam.
4th " Electrolysis of water.

OXYGEN

Preparation of Oxygen

Potassium chlorate is a white shining crystalline salt that is composed of one equivalent of potassium, one equivalent of Cl, and three equivalents of oxygen to the molecule, and has the formula $KClO_3$.

You see, this salt has a large proportion of O to the molecule, and in order to get this O in the form of a gas it would only be necessary to call to our aid some force capable of liberating it. The force that we apply is heat; but in order that we may lessen the amount of heat and get the same results, we mix with the $KClO_3$ about one-half its weight of MnO_2, which of itself does not change during the reaction, but comes out precisely as it went into the generating flask, its mere presence having reduced the amount of heat required.

Experiment 17: Carefully fill, over your pneumatic trough, five of your wide-mouthed bottles. Then weigh out on the trip scales ten grams of potassium chlorate and five grams of manganese dioxide, and having carefully mixed the same, place them in a test-tube which you have provided with a rubber stopper and a deliv-

ery-tube leading into a safety bottle, thus (teacher illustrates)! Pass the delivery-tube of the safety bottle beneath the mouth of your collecting bottles over the pneumatic trough, and heat your test-tube cautiously until the gas begins to come over rapidly. Fill all ~~five~~ of the bottles, ~~and having greased~~ a piece of your plate glass with tallow, slip beneath the mouth of each bottle, re-

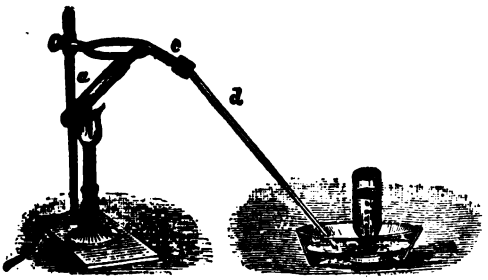

FIG. 5.

move from the pneumatic trough, and turn right side up and label, Nos. 1, 2, 3, 4, 5.

Tests: Into No. 1 place a piece of burning pine, and quickly cover up and stand aside.

Into No. 2 place a deflagrating spoon containing burning sulphur, cover quickly and stand aside.

Into No. 3 place a piece of phosphorus half the size of a pea, quickly cover and stand aside.

Into No. 4 place a piece of watch-spring previously prepared by wrapping around the end a bit of cotton string dipped into burning sulphur, cover and stand aside.

Into No. 5 lower a burning piece of charcoal. Cover and stand aside.

Now take bottle No. 1, and having added 5 c. c. of water, shake and test with litmus paper. Write results.

Do the same with each bottle and write out in full your explanations. Which litmus paper was affected?

Do you think these bottles contain alkalis? What effect has an alkali upon litmus? What effect has the liquid in these bottles upon litmus?

Try a few drops of each by adding to the test-tube one-quarter full of water into which you have placed one drop of methyl orange. Describe. Substances such as these that turn the vegetable colors opposite those produced by an alkali are called acid. An acid, then, has a sour taste, turns the vegetable colors opposite those colors produced by the alkalis, and always contains H. See text.

Percentage of Oxygen in Potassium Chlorate

Experiment 18: Clean and dry a porcelain crucible and heat it to redness for a few minutes in the flame of the burner. Cool and place in the crucible about ½ g. of manganese dioxide; weigh and place about 3 or 4 g. of potassium chlorate, mix carefully, and weigh again. Record all weighings. Now place the crucible on the *triangle* and heat over a low flame for fifteen minutes. Next heat more strongly for a few minutes. It is necessary to heat gently at first to avoid spattering. Cool, and weigh again. Heat again for a few minutes carefully and determine, by weighing, whether a constant weight has been reached. What does a constant weight here indicate?

Tabular Form

Weight of crucible plus MnO_2	
Weight of crucible plus MnO_2 plus $KClO_3$	
Weight of $KClO_3$	
Total weight after heating	
Weight of oxygen given off	
Percentage of oxygen	
Theoretical percentage of O in $KClO_3$	
Error	

Text, p. 119.

Percentage of Oxygen in the Air

Experiment 19: Place in a large test-tube about 1 g. of pyrogallic acid and dissolve it in 8 or 10 c. c. of water. In another large test-tube place an equal volume of strong potassium hydroxide solution. A third test-tube, small enough to be admitted, mouth downward, into the large test-tube, is fitted with a perforated rubber stopper.

Operations: Pour the potassium hydroxide solution into the pyrogallic acid solution and *immediately* drop the small test-tube, mouth downward (perforated stopper placed in small test-tube), into the mixture of alkali and pyrogallic acid. The alkaline pyrogallic acid will gradually absorb the oxygen from the volume of air in the small test-tube and the solution will rise in the small test-tube. Why? Allow to stand ten or fifteen minutes, agitating frequently. Remove the small test-tube by inverting the large one quickly over the sink. Mark on the small test-tube the volume of solution it contains. What

Tabular Form

BURETTE.

	First Reading.	Second Reading.	Difference.
For vol. of Nitrogen.			= vol. N.
For total vol. air.			= vol. air.
			vol. O.
			% of O.

does this volume represent? Mark also the position of the lower end of the rubber stopper. What does the volume of the tube to this point represent? Next, by admitting water from a burette, determine the total volume of air, volume of oxygen, and volume of nitrogen. Record in tabular form and compute the percentage of oxygen.

PROBLEMS

1. Find the weight of oxygen formed by heating 1080 g. of mercuric oxide. If one liter of oxygen weighs 1.4336 g., what volume of oxygen is formed by heating the above weight of mercuric oxide?

2. What weight of HgO is necessary to prepare 144 g. O?

3. (a) What weight of oxygen may be prepared from 367.5 g. of potassium chlorate? (b) What volume?

4. What weight of potassium chloride is formed by heating 367.5 g. of potassium chlorate?

5. What weight of potassium chlorate is necessary to use in preparing 10 g. of oxygen?

6. What weight of O can be obtained from 2610 g. of manganese dioxide? What volume?

7. What weight of Mn_3O_4 is formed by heating 2610 g. of MnO_2?

8. Find the percentage of O in HgO.

9. What is the percentage of oxygen in potassium chlorate?

10. Find the percentage of oxygen in manganese dioxide.

11. How much hydrogen would be liberated by dissolving 20 grams of magnesium in sulphuric acid? What volume?

12. How much sulphuric acid would be used in the preceding problem?

13. How much magnesium sulphate would be produced in the preceding?

14. If a liter of hydrogen weighs .09 of a gram, how much zinc necessary to liberate a cubic meter of hydrogen?

15. Sodium dissolves in HCl to form NaCl. How much HCl would dissolve 46 g. sodium?

16. How much NaCl would be produced in the preceding?

17. How much of manganese dioxide necessary to produce 10 grams of oxygen? (Remember that Mn_3O_4 is produced.)

18. Ten grams of zinc dissolve in acetic acid, $H(C_2H_3O_2)$, forming zinc acetate $Zn(C_2H_3O_2)_2$. How much acetic acid necessary?

19. In the preceding, how much zinc acetate would be formed?

20. In the preceding, how much hydrogen would be liberated?

21. What weight of potassium chlorate is necessary to furnish 10 liters of oxygen?

22. What weight of mercuric oxide is necessary to furnish 5 liters of oxygen?

23. What weight of manganese dioxide is necessary to furnish 6 liters of oxygen?

24. What does 1 c. c. of water weigh? What weight of oxygen may be obtained by electrolysis of 1 c. c. of water? What weight of hydrogen? Give equation.

25. What is the volume of oxygen obtained in Problem 24?

26. From the experiment of electrolysis of water, tell what volume of hydrogen is obtained at the same time the above volume of oxygen is obtained.

27. From Problems 5 and 6 tell what is the total volume of gases obtained by electrolyzing 1 c. c. of water.

SUPPLEMENTARY PROBLEMS IN HYDROGEN

1. What weight Zn for 10 liters hydrogen? (Note: 1 liter hydrogen weighs .09 g. Change to weight first.)

2. What weight H_2SO_4 for 10 liters hydrogen?

3. Using Fe and HCl ($Fe + 2HCl = FeCl_2 + H_2$), what weight of hydrogen from 560 g. Fe?

4. How many g. in 1 liter of water?

5. What is the volume of hydrogen obtained in 24?

WATER

Hydrogen by Electrolysis of Water

Proportion of Hydrogen to Oxygen

Experiment 20: Take the electrolysis apparatus (see p. 24), and having filled the tubes with water and inverted them, place them over the electrodes and turn on the current. Notice all that takes place. After the one test-tube is full of a gas, turn off the current and compare the amount of gas collected with the amount in the other tube. How do the two volumes compare? Take the filled test-tube, and having removed it from the p. t. quickly apply a lighted match. What do you think the gas is? Now turn on the current and collect this tube full again, leaving the other tube over the electrode as before. When this first tube is again full, what is the amount of gas in the second tube? Now remove the second tube, and having blown out the match, so that there still remains a bright spark upon it, quickly thrust the glowing match into the second tube. What takes place? This gas is oxygen. Now, you see, we have secured both these gases from water, in the proportion of two parts by volume of hydrogen to one part of oxygen. Hence water is not an element, as was supposed by the earlier chemists, but a compound. Could water be

produced from the two gases? How? (Teacher's experiment with eudiometer.)

By actually weighing the different elements that can be reduced to a gaseous condition, it is found that hydrogen is the lightest. Hence it is taken as the standard when comparing the weights of gases.

Volumetric Composition of Water Vapor

Experiment 21: *a. Apparatus:* Electrolysis apparatus and sulphuric acid wash-bottle for preparing pure dry hydrogen and oxygen in the electrolytic proportions. Steam-jacket, eudiometer tube connecting at its upper end with a capillary tube, and the capillary terminating in a three-way stop-cock. Above the stop-cock and connecting with it a funnel containing mercury. Leveling vessel connected by stout rubber hose with the lower end of the eudiometer. Induction coil. Flask of water for supplying the steam-jacket. Leveling vessel and eudiometer filled with mercury.

b. Diagram:

c. Operations: 1. First be sure that the electrolysis apparatus has been in action long enough to sweep out all the air, and that it is supplying a slow, steady stream of the dry detonating gas; i. e., electrolytic hydrogen and oxygen 2 to 1 by volume. The apparatus is arranged as shown in figure. The leveling vessel is so placed that the mercury is about 2 cm. below the top of the eudiometer. Water in the flask is also boiling evenly and steam passing regularly through the steam-jacket.

2. Raise the leveling vessel until the mercury fills the capillary, flows through the three-way stop-cock, so turned that the mercury passes through it to the side entrance, and the mercury just appears at the tip of the tube terminating the side entrance to the three-way stop-cock. Next

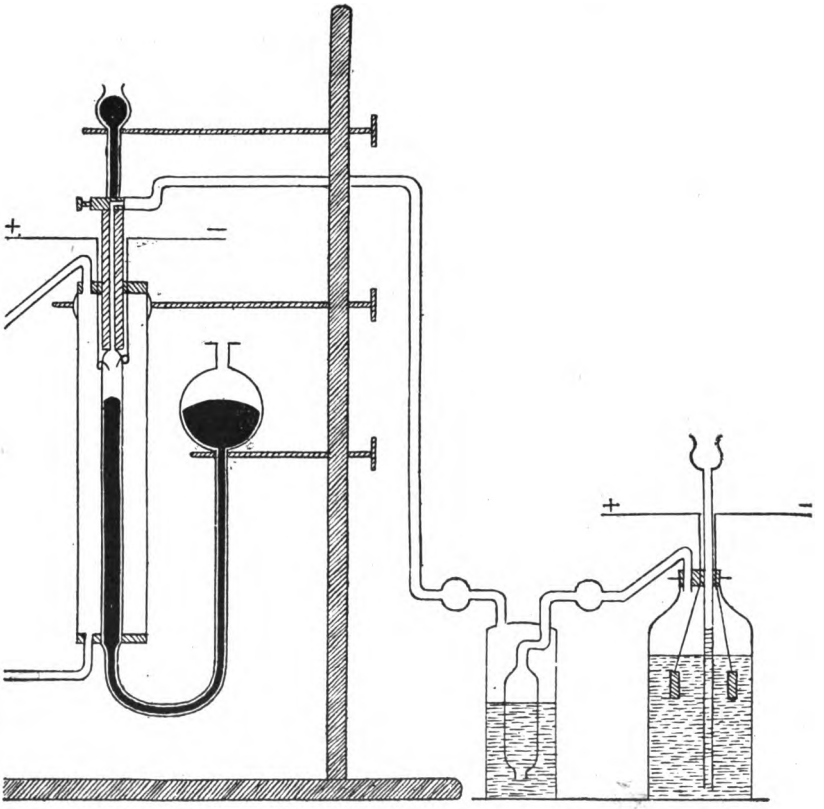

FIG. 6.

slip the delivery-tube of the electrolytic apparatus over the side tip of the three-way stop-cock and slowly lower the leveling vessel until about 6 c. c. of the electrolytic hydrogen and oxygen have been drawn into the eudiometer. Close the three-way stop-cock, disconnect the tube from the electrolysis apparatus, mark the exact depth of gas (H and O) in the eudiometer. Now cautiously turn the three-way stop-cock so that it will connect the capillary

with the mercury in the funnel above. A thread of mercury will pass down into the capillary. Now regulate the closing of the stop-cock so that the lower end of the thread of mercury will be 2 or 3 cm. above the eudiometer, but still in that portion of the capillary enclosed by the steam-jacket. This will prevent any of steam formed by the explosion of the hydrogen and oxygen from passing up into the cooler portions of the apparatus, condensing and so vitiating the result. Next lower the leveling vessel so as to expand the volume of the detonating gas, bringing it under reduced pressure and so lessening the danger of the explosion. Now pass the spark from the induction coil through the gas; a slight flash is the only evidence of the explosion. Raise the leveling vessel until the level of the mercury in it is the same as in the eudiometer. Note the volume of gas in the eudiometer. If the experiment has been carefully performed, the resulting volume of steam will be found to be exactly two-thirds the volume of mixed gases.

The preceding experiment in electrolysis of water shows that 2 volumes of hydrogen are produced for every 1 volume of oxygen. If, then, we used 6 c. c. of the mixed gases in the eudiometer, we had 4 c. c. of H and 2 c. c. of O to form 4 c. c. of water vapor. Or 2 volumes of hydrogen and 1 volume of oxygen give 2 volumes of water vapor. (Method of Dr. Geo. A. Hulett.)

Properties of Water

Vaporization and Condensation

Text, p. 38.

Experiment 22: Take your flask, and having filled it one-third full of water, place in the neck a stopper carrying a short delivery-tube bent at right angles. Boil the

water and notice the cloud of steam. What do you suppose fills the flask above the water after the boiling begins? Now hold a beaker filled with cold water above the delivery-tube and notice what accumulates on the outside of the beaker. Where does it come from? Now hold Bunsen flame immediately in front of your delivery-tube. What becomes of the visible vapor? Is true steam visible? What is it, then?

Freezing Point of Water

Text, p. 38.

Experiment 23: To find the freezing point of water, take a chemical thermometer and place it in a clamp; let the bulb dip into a beaker glass containing water. Now place your beaker within your water bath, and pack snow, or pounded ice, and salt, around the beaker. Then carefully stir the water within the beaker with a glass rod. When you see crystals beginning to form on the edge of the beaker note the reading of your thermometer. Why does this not always agree with the numbers in your text?

Boiling Point of Water

Text, p. 38.

Experiment 24: Again clamp your thermometer into place, with the bulb placed within your calorimeter about two-thirds filled with water (having your calorimeter placed upon the tripod). Now apply heat, carefully noting the temperature at which the large bubbles come to the surface and burst into steam. Why does this not agree with the number in the text?

What is meant by the dew-point? What are the drops of water that collect upon the cold surface of a pitcher of ice-water in the summer-time?

Now we wish to compare the weight of a given volume of water with that of some other substance, such as iron,

or lead; the ratio would represent how many times heavier one is than the other. That is, if we should weigh a cubic foot of iron and also a cubic foot of water, we would find the iron weighed nearly eight times as much as the water. This ratio is called the specific gravity of the iron. Water at 4° C. is always taken as the standard of specific gravity of solids and liquids. Why? Write out a law of your own for specific gravity.

SPECIFIC GRAVITY

Specific Gravity of Mercury

Experiment 25: Carefully weigh one of the small weighing flasks, and then fill with water and weigh again. Now remove the water, and having dried the flask, fill it with mercury and weigh again. Compute the specific gravity of the mercury. What did the difference in the two weights represent?

Method of Finding Specific Gravity

Experiment 26: Do the same as in the last experiment, only use the substance on the side table marked "unknown" instead of the mercury. What is the specific gravity of the "unknown"?

The method of finding the specific gravity of solids must be left for your course in physics. It is the province of chemistry to find the specific gravity of gases only. The lightest known gas, as already stated, is hydrogen, and is taken as the standard for the specific gravity of gases. A liter of this gas weighs .0896 g. What would be the specific gravity of O if a liter of it weighed 1.4336 g.?

In actual practice there are certain corrections to be made for temperature and pressure, which must be studied farther on in our course.

Examples: Suppose a given volume of H weighed x grams, and the same volume of N weighed y grams, what would be the vapor density of the N?

REVIEW

1. What is an element? Illustrate.
2. What is a compound? Illustrate.
3. What is a gas?
4. What gases have you experimented with?
5. What is specific gravity?
6. What is taken as the standard of specific gravity for solids and liquids?
7. What is taken as the standard of gases?
8. What is a chemical change? Illustrate.
9. What is a physical change? Illustrate.
10. What is an alkali? How formed?
11. What is its use?
12. Write an equation representing the action of K upon H_2O.
13. What is the substance formed when sulphuric acid acts upon zinc?
14. Write the equation representing the reaction.
15. What is an experiment?
16. Why performed?

When substances pass from a gaseous or liquid state to that of a solid, many of them form themselves into regular geometrical figures called crystals. Can you mention one such substance? What is the shape of the crystal of ice?

CRYSTALLIZATION

Crystallization of Potassium Dichromate

Keep the apparatus in the following experiments away from the dust.

Experiment 27: Take a test-tube and place therein a piece of potassium dichromate and fill the tube half full of water. Then boil over the Bunsen flame until the potassium dichromate is dissolved. Now pour in 10 c. c. of sulphuric acid and stand aside until the next laboratory period, then examine any solid that may be found in the bottom of the test-tube. Describe. Examine battery solution.

Crystallization of Alum

Experiment 28: Make a strong solution of alum by dissolving in boiling water in test-tube and stand aside for several days, then examine. Describe. Draw diagrams of each of the above forms of crystals.

Crystallization of Copper Sulphate

Experiment 29: Dissolve a piece of copper sulphate in 20 c. c. of water, and having placed it in your evaporating-dish, slowly evaporate one-half the liquid. Then stand aside until the next laboratory period. Examine. Explain.

Many substances crystallize only in the presence of water, and the water thus taken into the crystal is called water of crystallization.

Water of Crystallization

Action of Air and Moisture on Dry Substances

Text, p. 54.

(For definitions of Water of Crystallization, Deliquescence, Efflorescence, see text.)

Experiment 30: Place about 3 g. of copper sulphate in an ~~ignition~~ tube, incline the mouth slightly downward, and heat, noting the change in color. Cool, and transfer to a

test-tube. Add water. Note change in color and temperature. Use a thermometer to detect change in temperature.

a. Of what is this change characteristic?

b. What change usually occurs when a salt dissolves in water?

c. What is the cause of this change?

d. What is water of crystallization?

Experiment 31: Expose some crystals of calcium chloride, also of sodium sulphate, to the air for half an hour. What is deliquescence? What is efflorescence?

To Determine the Number of Molecules of Water of Crystallization in Copper Sulphate

Experiment 32: *a.* Clean and dry a porcelain crucible, then heat to redness and cool in a desiccator. Weigh accurately.

b. Crush and dry between filter papers about two grams of copper sulphate. Transfer to the crucible, and weigh.

c. Heat in the air bath at 220–240 degrees for one hour. Cool, and weigh again. Heat again to the same temperature for about twenty minutes. Then cool, and weigh again. If the weight has not remained constant to within .0005 g., repeat the heating until a constant weight is obtained. This caution is to insure that all the water be driven off.

d. Knowing the molecular weights of water and of copper sulphate, calculate the number of molecules of water of crystallization in copper sulphate. Record the data in tabular form.

Temperature by Centigrade and by Fahrenheit Scales

At what temperature does water begin to freeze or crystallize? How did you measure this temperature?

Compare your chemical thermometer with the thermometer at the side of the room. Do you notice any difference? What? How is a thermometer made? Compare the two principal kinds. Reduce the scale of one to that of the other. Suppose your room to be 70° F., what would it be by the C.? Suppose it to be 15° C., what would it be F.? Explain.

SOLUTIONS

Solution of a Solid in a Liquid

To Determine the Ratio between the Solubility of a Salt in Cold Water and its Solubility in Hot Water

In hot water:

Experiment 33: On a piece of paper weigh 10 g. of common salt. Put a third of this into 10 c. c. of distilled water contained in a large test-tube, heat to boiling, and keep just boiling until all is dissolved. Now carefully add a little more salt and boil as before. If the portion added dissolves, add another small portion, and continue the operation until no more will dissolve. Weigh the salt left on the paper. How much was dissolved? Set aside to cool. Why?

In cold water:

Experiment 34: While the solution is cooling, put into another 10 c. c. of water about one-half of what was left of the original 10 g. of salt. Cover the tube with the thumb and shake vigorously. If all dissolves, continue adding small quantities of salt as above directed until the last portion fails to dissolve. Weigh what is left. How much dissolved at the temperature of the room? Do not add large portions of salt when near the end of the operation, and also allow sufficient time for the liquid to dissolve all that it will.

Record results in the following tabular form:

Temp. of water.	Wt. of salt dissolved.	Vol. of water.	Ratio of weights.

Experiment 35: Weigh out 10 g. of potassium chlorate and proceed as above.

Experiment 36: Into 10 c. c. of water in a test-tube place a minute quantity of chalk and boil for a minute or more. What result?

Solution as an Aid in Chemical Action

Experiment 37: In a mortar rub together about 1 c. c. of dry tartaric acid and an equal volume of dry sodium carbonate. Is there any change? What is a mechanical mixture? Now take the same quantity of each substance, dissolve each separately in a test-tube in the least quantity of water, and then mix the two solutions in a beaker. What result? What is the difference between the behavior of the dry substances and that of those in solution?

Experiment 38: Repeat the operations, using one-half c. c. each of potassium iodide and lead nitrate.

Conclusion: State the use of water as a solvent in aiding chemical action.

(See Appendix, questions and answers.)

Change in Temperature during Solution

Experiment 39: Take 10 c. c. of water in a test-tube and ascertain its temperature. Add about 5 g. of pulverized potassium nitrate, stir quickly with a thermometer and note the greatest change in temperature. Repeat with copper sulphate. With granulated calcium chloride. With

potassium hydroxide. What is the cause of the different results?

Tabulate results:

Material.	No. degrees rise in temp.	No. degrees fall in temp.	Cause.

Solution of Liquids in a Liquid

Soluble in All Proportions

Experiment 40: To 10 c. c. of water in a test-tube add 15 c. c. of alcohol, taking care at first to add but a few drops at a time and shaking after each addition. What result? Finally add the rest of the 15 c. c. and shake. Result?

Soluble in Limited Proportions

Experiment 41: Repeat as above, using 10 c. c. of ether, instead of the alcohol, and shaking after the addition of each 5 drops. What result?

Solution of Gases in a Liquid

Effect of Pressure on the Amount of Air dissolved in Water. Apparatus provided

Experiment 42: Partially fill a filtering flask with fresh hydrant water and exhaust the air by means of an aspirator. Result? What is the relation between the solubility of gases and the pressure exerted upon them?

Effect of an Increased Temperature upon the Amount of Air (Gas) dissolved in Water

Experiment 43: On a wire netting covering the ring of the retort-stand place a beaker half full of hydrant

water and heat gently. Note that bubbles of gas pass off before the water reaches the boiling-point. What gas? What is relationship between the solubility of gases in a liquid and the temperature? How does this differ from your observation made in regard to the solubility of solids in a liquid?

Definition.—A solution is any homogeneous mixture the constituents of which cannot be separated by mechanical means.

SEPARATION OF SOLIDS FROM LIQUIDS

By Filtration

(For manipulation of the filter paper, consult instructor.)

Experiment 44: Take a gram of powdered chalk suspended in 20 c. c. of water, and pour this on the filter. Note whether the liquid which passes through is clear. That which passes through is called the filtrate, and that which remains on the filter paper, the precipitate.

By Distillation

Experiment 45: The apparatus provided for this experiment represents the ordinary distilling outfit. It consists of flask, thermometer, condenser, and receiver. The liquid in the flask is boiled. The vapor is condensed by a stream of cold water, which is admitted between the jacket and the inner tube of the condenser at the lower opening, and which escapes through the upper opening into the sink.

In the distilling flask place 100 c. c. of hydrant water. Arrange the condenser and connect it with the flask, and, having placed your flask upon a wire gauze on your ring stand, boil the water until two-thirds of it has passed

over, collecting the distillate in a clean beaker glass, and noting the temperature of the escaping steam.

After most of the liquid has been distilled, what remains in the distilling-flask? What effect does this have on the water? Take about 5 c. c. of the distillate and evaporate it to dryness over the open flame. Heat very gently at last, noting the residue, if any.

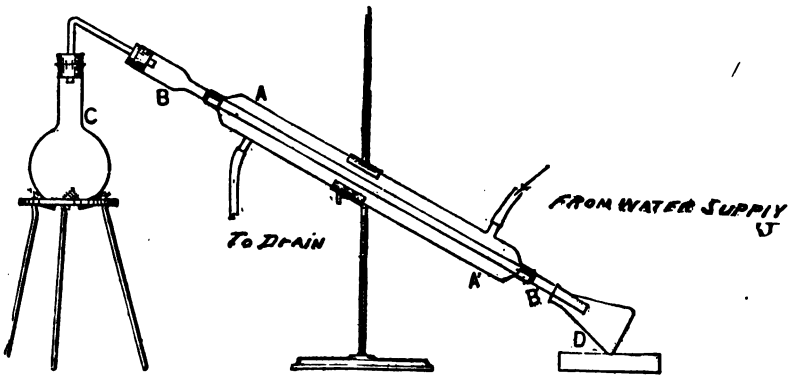

FIG. 7.—Condenser arranged for the purification of water by distillation.

By Crystallization

Concentrate a solution of potassium chloride and sodium nitrate.

By Precipitation

Experiment 46: Dissolve all that will of 5 g. of potassium sulphate in 10 c. c. of cold water. Add 5 c. c. of alcohol. What result? Compare the precipitate with the original substance. What was the cause of the precipitation?

Give a summary of the methods of separating solids from liquids.

What is the appearance of the water in the flask? What was the object of boiling it and then condensing the vapor? What is the difference between hard and soft water? Why is rain-water soft? Which is the more nearly pure, the rain at the beginning of a rain storm or that at the close? Why? Why do you use soap when washing? Which takes less soap, hard water or soft water? Why?

CHLORINE

Preparation of Chlorine

UNDER HOOD. 17.

Experiment 47: Place 9 c. c. of water and 1 c. c. of concentrated hydrochloric acid into a 20 c. c. test-tube, and cool to temperature of hydrant-water. Add a *very little* lead dioxide from the corner of a glass spatula, adding slowly until the merest trace remains undissolved. Change of color in liquid? Odor? Now boil the solution, holding moist blue litmus paper over the mouth of the tube. Why? Odor? Why? When the solution is nearly colorless, set aside to cool. Why? Explain the change that takes place at the two temperatures.

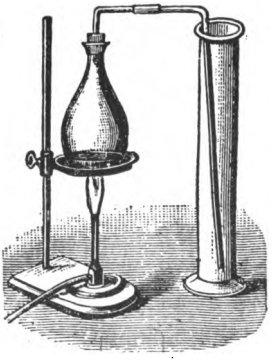

FIG. 8.

Experiment 48: Repeat, using 2 c. c. of water to one of acid, and manganese dioxide in the place of the lead dioxide. After having boiled the mixture, the solution may be thrown away without waiting to cool it.

Experiment 49: To 75 c. c. of hydrant-water in a beaker add slowly 75 c. c. of concentrated sulphuric acid, and mix well. While this is cooling, prepare a generating

apparatus, as shown in Fig. 8. Mix on a large sheet of paper 50 g. each of sodium chloride and of manganese dioxide, place in a flask, add the cool, diluted sulphuric acid through the safety tube. Heat but gently. In dry bottles, collect enough gas by upward displacement of air for Experiment 50, and with the remainder of the gas saturate 25 c. c. of water, and reserve the solution for future use.

What weight of each of the compounds used in the experiment for the preparation of chlorine is required to yield one liter of gas at the standard conditions?

Tests for Chlorine

UNDER HOOD.

Experiment 50: Introduce into the bottles:

a. A piece of dry calico. After a few moments moisten it and introduce again. Repeat, using a strip of litmus paper.

b. A *very little* finely powdered antimony from a glass spatula. (This bottle is easily cleaned afterward with a little concentrated hydrochloric acid.)

c. A piece of copper foil.

d. A piece of phosphorus as large as a pea.

e. Invert a bottle of dry chlorine over water and let it stand for some time. Compare the ratio of solubility of chlorine with that of hydrochloric acid.

f. (Teacher): Light a jet of hydrogen and introduce it into a bottle of chlorine. Hold in it a glass rod moistened with silver nitrate solution. Hold a strip of moist litmus paper in the mouth of the bottle. Compare this effect with that of chlorine on litmus. What is the resemblance between the chemical action of chlorine and that of oxygen?

Hydrochloric Acid

UNDER HOOD: Teacher illustrates.

Text, p 88.

Experiment 51: Arrange a generating apparatus as in Fig. 4.

Place in generating flask 25 g. of sodium chloride, pour upon it 50 c. c. of dilute sulphuric acid (two volumes of acid to one of water), and heat gently.

Collect four bottles of the gas by downward displacement of air, covering them with greased glass plates as soon as filled. In order that you may tell when the bottle is filled, hold above the bottle a piece of moist blue litmus paper; this will enable you to tell when it is full. Why? Collect the remainder of the gas in a beaker of water.

Tests with Hydrochloric Acid

Experiment 52: Invert one of your bottles of gas in a beaker half full of water. Explain. Taste a drop of the liquid in the beaker soon after.

Blow the breath across the mouth of the second jar. Insert into another jar a burning splinter. What properties of HCl are shown by these experiments?

Density of a Saturated Solution of Hydrochloric Acid

Experiment 53: Place in a weighed test-tube about 5 c. c. distilled water and determine its weight exactly. Mark the depth of water with a label. Next set the test-tube in a dish of cold water or ice and pass hydrochloric acid gas through it for about five minutes, keeping cold all the time. Now remove the delivery-tube of the hydrochloric acid generator; note from the level of the liquid whether the volume of this solution is the same as that of the original volume of water. If not the same, mark this

new level and weigh the solution. What is the weight of the hydrochloric solution?

Now pour out the solution and fill the test-tube with distilled water to the depth marked by the solution. Weigh again. What is this weight of water? What was the weight of the equal volume of the hydrochloric acid solution?

Compute the density of this hydrochloric acid solution. Tabulate results as follows:

Wt. of test-tube.	Wt. of test-tube and water.	Wt. of water.	Wt. of HCl solution.	Wt. of HCl absorbed by water.	Wt. of the vol. water = vol. HCl sol.	Density of HCl sol.

Electrolysis of Hydrochloric Acid

Experiment 54: Take the battery and electrolysis apparatus just as in the decomposition of water, only having the platinum electrodes moved higher up in the tubes, and having within the jar a saturated solution of sodium chloride, to which has been added one-tenth of its volume of a saturated solution of hydrogen chloride in water. (To make the saturated solution of hydrogen chloride, make the gas as in Experiment 51. Here the wash-bottle of H_2SO_4 is not necessary.) Then pass the gas from the delivery-tube into a beaker glass filled with water until the water is saturated. When the current is turned on, a gas will be evolved at the negative pole of the battery. Invert over the electrode a test-tube filled with H_2O. When the tube at this pole is full of gas, remove and test for hydrogen. The gas at the other pole will not appear for some time, but should the battery run long enough a gas would appear there. This gas will be green

in color, and its volume will equal the volume of H given off in the same time, and have a very disagreeable odor. It is called chlorine. Why do you think it did not begin to appear at the same time with the H? Should the apparatus run long enough, equal volumes of the two gases would be obtained in equal periods of time. What does this prove to you?

Now that you have made an alkali and an acid, we will unite the two and see what form of compound is produced. What alkali did you make? How? What acid did you make? How? What is an alkali? What is an acid? Name and write the symbols to all the acids that you can.

Preparation of Sodium Amalgam

UNDER HOOD: Four work together.

Experiment 55: Place 250 g. of clean dry mercury in a Hessian crucible, cover with a piece of sheet iron, cut 2.5 g. of clean sodium into pieces the size of a small marble, and place them in the crucible. To start the reaction, heat three or four grams of mercury in a test-tube, slightly raise the cover of the crucible, pour in the heated mercury, and instantly remove the hand. A reaction accompanied by a flash of light will occur. Never spill mercury in the sink.

Stand back, so as not to inhale any fumes of mercury. When the reaction is finished, stir the amalgam with an iron wire, allow to cool, and place in a stoppered wide-mouthed bottle and label "Sodium Amalgam."

Relation Between Volume of Hydrogen and Volume of Hydrochloric Acid

(*Teacher's Experiment*)

Experiment 56: Take a combustion tube and place a rubber band or string 15 c. c. of the distance from the

sealed end, and another just half-way between the first rubber band and the open end of the tube. Have ready a small glass tube sealed at one end that will hold 18 c. c. of sodium amalgam and completely fill the same. Now completely fill the large tube with dry hydrogen chloride, displacing the air as in the case of collecting the gas in the previous experiments. When the tube is filled with gas, pour in the sodium amalgam, quickly cover the mouth with the thumb, and bring every particle of the gas into contact with the amalgam, by repeatedly inverting the tube. Finally, place the open end of the tube in a battery jar filled with water and remove the thumb. Lower the tube into the jar so the level of the water is the same within and without.

What is the relation of the volume of the gas remaining to that which was originally contained in the tube? Now, again place the thumb over the mouth of the tube and take the tube from the water, invert it, remove the thumb, and instantly apply the flame of a match.

What decomposes the hydrogen chloride? What became of the Cl gas? Is hydrogen chloride a gas or a liquid? What is the liquid on your work-table which is labeled hydrochloric acid? Could you decompose hydrogen chloride by means of the electric current? Explain. Could hydrogen chloride be formed by the union of the two gases, hydrogen and chlorine? How much heat was given off in the decomposition? See work in energy. Explain.

Conclusion: From Experiments 53 and 56 give complete volumetric composition of hydrochloric acid gas.

ELECTROLYSIS

Statement of Facts

(To be amplified from text)

1. As has been illustrated (p. 37), the electric current can produce chemical separation for substances in solution. The process is called electrolysis, and the substance separated is called electrolyte. (Read Appendix on Ionization.)

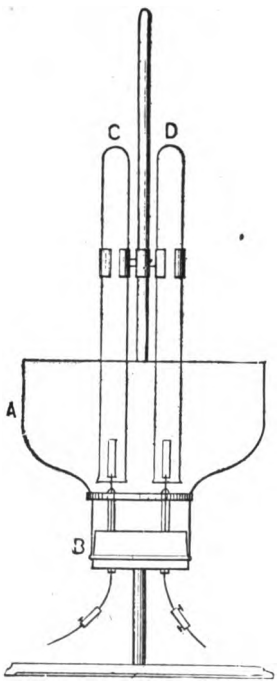

FIG. 9.—Apparatus for the electrolysis of water.

2. The electrolyte always consists of a metal, or else hydrogen joined to a negative ion or radical, as oxygen, chlorine, the NO_3, SO_4 ion, etc. Hence it is a chemical salt.

3. The metal or hydrogen of the electrolyte always has a charge of positive electricity, and the negative ion has a charge of negative electricity.

4. By a law of electricity there is an attraction between opposite electrifications. Hence the metal or hydrogen with its positive charge goes to the negative electrode in electrolysis, and the negative ion with its negative charge goes to the positive electrode. The apparatus, with contents for the electrolysis, is called the electrolytic cell. The wires from the battery furnishing the current terminate in strips of *platinum foil* inside the cell. These terminations are called the electrodes.

Draw diagram of apparatus.

Electrolysis of Water

Experiment 57: *a.* Pure water is almost a non-conductor of electricity, but water containing a little sulphuric acid is a good conductor. Sulphuric acid in water becomes largely dissociated into H_2 ions carrying positive charges, and SO_4 ions carrying negative charges.

b. The current passing through the solution directs the positive H_2 to the negative electrode, where it collects as a gas.

c. At the same time, the current directs the negative SO_4 ions to the positive electrode. The SO_4, however, seems to be incapable of separate existence except as an ion; the SO_4 at the negative electrode decomposes water, forming oxygen and sulphuric acid, according to the equation, $SO_4 + H_2O = H_2SO_4 + O$.

d. Observe that it is water which is eventually decomposed, and that the amount of sulphuric acid remains constant. Note here the volumes of oxygen and hydrogen obtained, and test each.

Electrolysis of Copper Sulphate

Experiment 58: Copper sulphate in solution is largely dissociated into Cu and SO_4 ions. The electric current directs the positive copper to the negative electrode, where it collects as a copper plate. The SO_4 is simultaneously directed to the positive electrode. There the SO_4 decomposes water, forming sulphuric acid and oxygen.

Equation: $SO_4 + H_2O = H_2SO_4 + O$.

Electrolysis of Potassium Sulphate

Experiment 59: Potassium sulphate in water solution is largely dissociated into potassium ions carrying positive charges and SO_4 ions carrying negative charges of

electricity. The current directs the potassium ions to the negative electrode, but, as we know, the potassium cannot exist as the free element in the presence of water except in the condition of an ion. The potassium at this point, then, decomposes water, forming potassium hydroxide and free hydrogen.

Equation: $K + H_2O = KOH + H$.

As in the electrolysis of copper sulphate, the SO_4 ions are directed to the positive electrode, where they decompose water, forming sulphuric acid and free oxygen. Note tests for each.

PREPARATION OF SOLUTIONS

Standard Solutions

A solution of hydrochloric acid, containing 36.4 g. of acid per liter, contains 1 g. of acid hydrogen per liter; a solution of sulphuric acid containing 98 g. (molecular weight in grams; molecular mass, see text, p. 227) of acid per liter contains 2 g. of acid hydrogen per liter, or, if containing 49 g. per liter, contains 1 g. of acid hydrogen per liter. Solutions containing 40 g. of sodium hydroxide or 56 g. of potassium hydroxide per liter contain respectively 23 g. of sodium and 39 g. of potassium per liter.

Definition.—A solution containing 1 g. of acid hydrogen, no matter of what acid, per liter, is called a Normal Standard Solution (N. S. S.) of acid. Solutions containing 23 g. of sodium or 39 g. of potassium per liter are called Normal Standard Solutions of alkalis. Solutions containing one-tenth the above amounts per liter are called deci-normal, N/10, solutions. Hence, to make a normal solution of potassium hydroxide, place 56 g. of the solid KOH in a liter flask, and fill the flask with distilled water to the liter mark.

Neutralization

To Prepare Deci-normal Solutions of Alkalis and to Determine the Weight of Sulphuric Acid Neutralized by a Given Weight of Alkali

Experiment 59: *a.* Potassium hydroxide solutions on the side table contain 56 g. of alkali per liter. From the alkali burette on the side table draw out into a clean bottle exactly 20 c. c. of the alkali. Add enough distilled water to make it a deci-normal solution. Question: To what volume must it be diluted to reduce it to a deci-normal solution? Next draw off 10 c. c. of the sulphuric acid solution containing 49 g. per liter and dilute to 100 c. c. Measure out distilled water in the graduate.

b. Place your acid in your left-hand burette and alkali in the right-hand burette. Draw off into a beaker exactly 10 c. c. of acid, and add 5 or 6 drops of phenol-phthalein and neutralize with the alkali, noting the exact amount of alkali used.

c. Computations. 1. Knowing that the original solution on side table contains 56 g. of alkali per liter, compute the weight of alkali used to neutralize the 10 c. c. of your acid solution. 2. Compute the weight of acid in 10 c. c. of your solution. What is the ratio between the weight of acid used and the weight of alkali necessary to neutralize it? What is the theoretical ratio?

Record results in the following tabular form:

Vol. acid sol.	Name of alkali.	Vol. alkali used.	Wt. of alkali used.	Wt. of H_2SO_4 used.	Wt. of HCl used.	Ratio of weights alk: H_2SO_4.	Ratio of weights alk: HCl.

Ratio of weight of potassium hydroxide to sodium hydroxide.
Ratio of mol. wt. of potassium hydroxide to sodium hydroxide.
Ratio of strength of potassium hydroxide to sodium hydroxide.

Experiment 60: Proceed in exactly the same way with the sodium hydroxide solution, which contains 40 g. of alkali per liter.

Prepare a deci-normal solution of hydrochloric acid. The side table solution contains 36.4 g. of hydrochloric acid per liter of solution.

Titration

Sodium Chloride and Sulphuric Acid

Experiment 61: Take your flask, and having washed it thoroughly with hot water, and rinsed with distilled water, place therein 15 grams of sodium chloride. Now make a mixture of 22½ c. c. of sulphuric acid and 80 c. c. of distilled water, and having allowed the mixture to cool thoroughly, pour it upon the sodium chloride in the flask. Arrange the Liebig condenser so that it terminates in a pair of ordinary wash-bottles, each bottle containing 10 c. c. of distilled water. Now boil the liquid in the flask until one-half of the liquid has passed over. Allow it to cool thoroughly, and again add 80 c. c. of distilled water and boil again until one-half has passed over. All the hydrochloric acid will now have passed over with the distillate. Take the contents of the wash-bottles, carefully pour them into a clean cylinder, and, having measured, pour into a perfectly clean bottle and dilute to 250 c. c. with distilled water. The 15 g. of sodium chloride should produce 9.18 g. of hydrochloric acid, therefore the 250 c. c. contain the 9.18 g. of hydrochloric acid. This is a normal standard solution of HCl. Titrate with KOH to test the HCl which you have made. Write the chemical equations for the reactions involved, both in preparing the hydrochloric acid and in the titration.

Sodium Carbonate and Hydrochloric Acid

Experiment 62: Fill your burette with the standard solution of hydrochloric acid, and having taken 25 c. c. of the standard solution of sodium carbonate into which you have placed two or three drops of methyl orange, run the acid from the burette until the pink color is reached. Measure and estimate your results. What is a standard solution? Solutions of known strength used in volumetric analysis are called standard solutions.

When the solution is of such strength that the particular volume which contains one gram of acid H, 23 g. of Na, is one liter, then the solution is known as a normal standard solution.

PROBLEMS

Determining Concentrations of Solutions

1. A potassium hydroxide solution contains 20 g. of alkali per liter. What weight of sulphuric acid will be neutralized by 100 c. c. of this solution? Give equation.

2. Give the equation for forming potassium primary sulphate. What weight of $KHSO_4$ will be formed by 200 c. c. of the above KOH solution?

3. A sodium hydroxide solution contains 20 g. of alkali per liter. What weight of sulphuric acid will be neutralized by 100 c. c. of the solution? Give equation.

4. What weight of NaOH will be used in changing 100 g. of H_3PO_4 to NaH_2PO_4? Give equation.

5. What weight of sodium hydroxide will be used in changing 100 g. of phosphoric acid to sodium secondary phosphate? Give equation.

6. What weight of potassium hydroxide and sodium hydroxide each will be used in the changing of 100 g. of phosphoric acid to potassium sodium hydrogen phosphate? Give equation.

7. Using calcium hydroxide, write equations for form-

ing the primary, secondary, and normal salts of phosphoric acid.

8. Twenty-five c. c. of N/10 sulphuric acid neutralize 40 c. c. of sodium hydroxide of unknown strength. Find the absolute weight of sulphuric acid in the 25 c. c. used. From the equation for neutralization, $H_2SO_4 + 2NaOH = Na_2SO_4 + 2H_2O$, find the weight of alkali neutralized by this amount of acid. Noting that the weight of alkali found is contained in 40 c. c., determine the weight of alkali per liter.

9. Sixty c. c. of N. S. S. potassium hydroxide neutralize 80 c. c. hydrochloric acid of unknown strength. Find the weight of acid contained in one liter of the solution. To what volume would it be necessary to dilute a liter of this hydrochloric acid to reduce it to deci-normal strength?

10. Fifty c. c. N/10 sulphuric acid neutralize 15 c. c. of ammonia solution. $2NH_3 + H_2SO_4 = (NH_4)_2SO_4$. Find the weight of ammonia per liter. How much more ammonia gas must be passed into the solution to bring it up to N. S. S. strength (up to 17 g. per liter)?

The names and symbols of the following acids should be committed to memory:

HNO_3	Nitric.	HCl	Hydrochloric.
HNO_2	Nitrous.	HF	Hydrofluoric.
H_2SO_4	Sulphuric.	HI	Hydriodic.
H_2SO_3	Sulphurous.	HBr	Hydrobromic.
$H_2S_2O_3$	Thiosulphuric.	HPO_3	Metaphosphoric.
HClO	Hypochlorous.	H_3PO_2	Hypophosphorous.
$HClO_2$	Chlorous.	H_3PO_3	Phosphorous.
$HClO_3$	Chloric.	H_3PO_4	Phosphoric (ortho).
$HClO_4$	Perchloric.	$H_4P_2O_7$	Pyrophosphoric.
$H_2C_2O_4$	Oxalic.	H_2CO_3	Carbonic.
$H(C_2H_3O_2)$	Acetic.	$H_2C_4H_4O_6$	Tartaric.

Write salts of each of the above-named acids.

NITROGEN

Preparation of Ammonia Gas

Experiment 63: Place a mixture of 10 g. quicklime and 8 g. ammonium chloride in a flask.

$$CaO + 2NH_4Cl = CaCl_2 + 2NH_3 + H_2O.$$

Lime — sal. ammoniac — calcium chloride — ammonia.

The mixture is heated, and the gas led off by a delivery-tube. Ammonia gas cannot be collected over water because of its great solubility; but it can be collected over mercury, or, more simply, by upward displacement.

Tests: To ascertain when the jar is filled with ammonia, it is necessary only to hold a stopper or glass rod moistened with hydrochloric acid below the mouth of the bottle; and if the jar is full, and ammonia gas is escaping, dense white fumes of sal. ammoniac will be seen to form round the stopper.

$$NH_3 + HCl = NH_4Cl$$

Place a jar of ammonia, mouth downward, in water. Result? Determine whether it is combustible and a supporter of combustion.

Formation of an Ammonium Salt

Experiment 64: Place the ammonia solution obtained in experiment in a small evaporating-dish, and add dilute sulphuric acid from a burette until the solution is neutral. Why? (Use litmus paper.) Evaporate the solution to saturation and set aside to cool. UNDER HOOD. Dry a portion of the crystals and heat them over the free flame in a porcelain crucible, and test the escaping fumes from time to time with litmus paper. Why? Upon another portion, in a small beaker, pour 1 c. c. of potassium hydroxide solution, cover the beaker with a clean watch-

glass, on the under side of which a strip of moist red litmus paper has been stuck, and allow it to stand. Why? Over the mouth of the ammonia bottle hold a glass rod dipped in hydrochloric acid. Why?

Volumetric Composition of Ammonium Chloride

Experiment 65: This apparatus consists of two tubes, each 20 c. m. in length and 5 c. m. in width, connected by means of a rubber tube (provided). Close the middle of the rubber tube with a pinch-cock, fill the tubes with mercury (very carefully dried) and invert them over a mercury trough, side by side. Fill one tube with pure *dry* ammonia gas, and the other with pure *dry* hydrochloric acid. Now remove the pinch-cock between the two tubes and allow to stand for a few minutes. Why?

Volumetric Composition of Ammonia Gas

Experiment 66: *Apparatus.*—Mercury, mercury-bath, eudiometer tube, ammonia gas generator and drying appa-

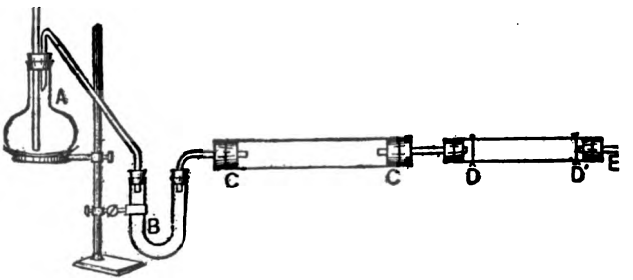

FIG. 10.—A, generating flask. B, U-tube containing quicklime. C C, tube filled with quicklime. D D, tube filled with soda-lime.

ratus (How can you dry ammonia gas?), static machine, battery or current from dynamo, meter stick, thermometer and barometer.

Operation.—Fill the eudiometer tube with clean, dry mercury, and mount in mercury-bath and clamp in position. Generate ammonia and dry in drying tubes, as shown in following diagram, Fig. 10.

Pass the ammonia gas through the drying apparatus long enough to make sure that all the air has been expelled, then introduce about 4 c. c. of the gas into the eudiometer. Place a thermometer so that the bulb dips into the mercury, and allow the whole apparatus to stand twenty minutes. Then make the readings.

Use of Chlorine in determining the Volumetric Composition of Ammonia

Relation by Volume Between Nitrogen and Hydrogen in Ammonia

Experiment 67: Close at one end a glass tube about 50 cm. long, and fill it with a saturated solution of salt and water. Invert the tube over a beaker containing some of the same solution and place the whole apparatus under the hood. Set up a chlorine generator. When the air is expelled from the apparatus, fill the glass tube with chlorine by displacement of the salt solution. Place the finger over the end of the tube and transfer it to a beaker of ammonium hydroxide. The chlorine comes in contact with an ammonia gas, so that the reaction may be considered between chlorine and ammonia. As soon as the ammonium hydroxide has risen about two inches in the tube, transfer to a tall receiver containing water. When the reaction is complete and all the fumes dissolved, measure the depth of gas remaining in the tube, and also the total length of the tube. If much ammonia has been used, it may be necessary to invert the tube over dilute sulphuric acid until the excess of ammonia has been absorbed.

For the note-book:

1. Name or purpose of the experiment.
2. Materials and apparatus.
3. Operations and observations. Give a brief account of the method. (a) With what did the chlorine unite? Give equation. (b) With how much did the chlorine unite? (c) From what was the element obtained? (d) What element from the ammonia compound remains, and how much of it?
4. From the preceding, sum up the proof for the volumetric composition of ammonia.

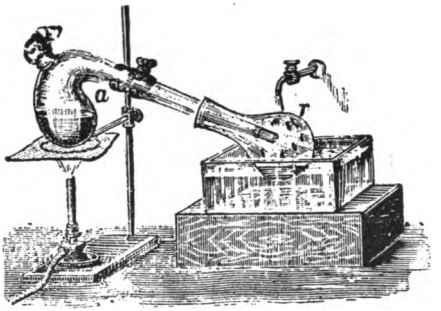

FIG. 11.

Nitric Acid

Experiment 68: Place in your retort 10 g. of sodium nitrate and 30 c. c. of H_2SO_4. Place the neck of the retort within the neck of the receiver, over which cold water is allowed to run continually. After heating twenty-five minutes, remove the flame, take the retort neck from the receiver, and pour out the condensed acid from the receiver into a well stoppered bottle, label, and keep for future use.

What is the color of your acid? What would be its color if pure? What is its formula? What is its radical?

What salt did you take in its preparation? Could you have taken any other salt? What?

Give the properties of HNO_3. Give its molecular weight.

Give the properties of HCl. From what did you make the HCl?

Give its molecular weight. Suppose you mix the two acids, what would you have? What would be the best proportions in which to mix them? For what is the mixture used? How could you make an alkali? What alkalis?

What would be formed by adding HNO_3 to NaOH?

Pulverize a few grains of charcoal and heat it; upon the heated charcoal pour a little strong HNO_3. Explain.

Nitric Oxide

Experiment 69: Into a test-tube put a few bits of copper and cover them with HNO_3. Notice all that takes place, and describe. What salt have you? What gas was given off? The equation for the reaction is as follows:

$$3Cu + 8HNO_3 = 3Cu(NO_3)_2 + 4H_2O + 2NO$$

Proof for the Presence of Hydrogen, Nitrogen, and Oxygen in Nitric Acid *

Experiment 70: *a. Hydrogen.*—Test a dilute solution of nitric acid with litmus. The reddening of blue litmus is the proof for the presence of the hydrogen ion.

b. Nitrogen.—Place a strip of metallic aluminium in a test-tube with about 5 c. c. sodium hydroxide solution. Warm until hydrogen begins to be evolved, then add a few drops of nitric acid, and test the gas at the mouth of the test-tube with reddened litmus paper. The litmus is turned blue, showing that ammonia gas is given off.

* Method of Mr. B. W. Peet.

The nitrogen of the ammonia is obtained from the nitric acid.

c. Oxygen.—Take a piece of ferrous sulphide the size of a pea, dissolve in dilute hydrochloric acid, and divide the solution into two portions. What does the solution now contain? Complete the equation $FeS + 2HCl. =$.

To the first portion add a little barium chloride solution. Result?

To the second portion of the solution add one-half c. c. of HNO_3, boil, then add barium chloride. Result? The nitric acid transforms the original solution of ferrous chloride and hydrogen sulphide into a solution of sulphuric acid and ferric sulphate by adding oxygen to the original substances. This proves that there is oxygen in nitric acid. Barium chloride with these products forms the heavy white, insoluble barium sulphate. This could not have been formed unless oxygen had been added to materials originally in the solution. What test proves this?

Union by Volume.—Nitric Oxide with Oxygen

Experiment 71: Into a receiver containing 25 c. c. of oxygen decant 25 c. c. of NO gas. Let in a few bubbles at a time, and gently agitate until the reddish brown fumes of NO gas are dissolved in the water. The volume of oxygen remaining should be just half the original volume. Now decant another 25 c. c. of NO gas into the remaining O; and if both gases are pure, all the O will have united with NO, forming NO_2, which is soluble in water, and two volumes of NO have united with one of O to form NO_2.

The NO gas to be pure should be made by heating HNO_3 with $FeSO_4$. Test-tubes graduated on the surface with a file may be used as receivers for the two gases.

The NO unites with one O from the air and forms NO_2. This is what gives the brown gas.

Brown Ring Test for Nitric Acid

Experiment 72: Make a fresh solution of $FeSO_4$ by dissolving a small crystal of the $FeSO_4$ in moderately warm water, add a few drops of any solution containing a nitrate; e. g., sodium nitrate. After the mixture is cool let trickle down the inside of the test-tube a few drops of H_2SO_4 and notice what forms within the solution.

Separation of Silver from Copper in a Silver Coin.—Deposition of Silver on Copper

Experiment 73: Place a dime that has been carefully weighed in the bottom of your evaporating-dish, and then pour over it 5 c. c. of HNO_3 (UNDER HOOD), and allow it to react for half a minute. Then remove the dime and wash with water, so as not to injure the coin; dry, and weigh again. Now take the liquid in the evaporating-dish and add 10 c. c. of water and slowly evaporate to dryness. What causes the blue color in the dish? What fractional part of a coin is copper? Now, again dissolve the residue in the evaporating-dish in 10 c. c. of water and place in a test-tube and place therein a bright copper wire. Describe. After the reaction ceases, pour upon a filter paper and wash with cold water. What have you in the liquid? What have you upon the filter paper? Dissolve this residue in HNO_3, evaporate to dryness, add 20 c. c. of water. What have you in solution? Add a few drops of HCl. After the precipitate ceases to form, filter, dry the residue, and weigh. Compare the amount of silver in the silver chloride with the original amount of silver taken from the coin. What is the process called?

Take the filtrate and place therein a strip of lead. Again filter, and add to the filtrate a strip of zinc. Explain.

Preparation of Nitrous Oxide

Experiment 74: Place about 20 grams of dry nitrate of ammonium in a flask furnished with a wide delivery-tube, as used in the preparation of oxygen. Heat the salt until it begins to decompose, and then regulate the flame of the lamp; otherwise the gas is evolved too rapidly. The pneumatic trough must be filled with warm water, because the gas dissolves considerably in cold, but to a less extent in hot water.

Tests: Collect a test-tubeful of the gas, and insert a glowing chip; it will at once burst into flame, just as in the case of oxygen.

Having collected several jars of the gas, proceed as with oxygen, to burn phosphorus in a deflagrating spoon; the phosphorus will burn almost as brightly as in oxygen.

If a piece of sulphur, well alight, is introduced into the gas, it continues to burn almost as brightly as in oxygen, but if only feebly burning, the flame is put out. The reason of this is that the gas must first be decomposed into its constituent elements, nitrogen and oxygen, before it can act as a supporter of combustion, and to effect this decomposition a tolerably high temperature is necessary.

Charcoal also burns as in oxygen. Even a steel watch-spring burns as in oxygen, if tipped with brightly burning sulphur.

SULPHUR

Study of the Reactions Between SO_2, H_2O, and HNO_3

Experiment 75: *a.* Burn some sulphur in a bottle containing a little water; agitate. Give equations. Divide into two parts; test one part with barium chloride. What form of ppt.? Give equation.

b. 1. To the remainder of the sulphurous acid and some nitric acid, and boil.

2. Now test with barium chloride, and compare the ppt. with that obtained before boiling with nitric.

(a) $H_2SO_3 + 2HNO_3 = H_2SO_4 + 2NO_2 + H_2O$.

(b) Give equation.

c. Proof that NO_2 is formed:

Pour a little nitric acid into a dry bottle and agitate. What colored fumes appear? Give equation.

d. Can NO_2 be further reduced?

1. Half fill a bottle with NO_2 gas. To get this gas into the bottle, put a small piece of copper into a test-tube, add a little nitric acid, and lower into the bottle. Care must be observed not to get too much of the gas.

2. Next burn some sulphur in the bottle. What result? Give equation.

(a) $Cu + 4HNO_3 = Cu(NO_3)_2 + 2NO_2 + 2H_2O$.

Hydrogen Sulphide

Experiment 76: *a. Preparation* (Hood).—Prepare a gas generator as in Fig. 5, using nothing larger than a 100 c. c. flask, and connnect this with a wash-bottle containing about 30 c. c. of water. Place in the flask some ferrous sulphide, and pour upon it 10 c. c. of dilute sulphuric acid (1 to 3). Collect two bottles of the gas by displacement of air.

b. Properties (Hood).—Moisten a strip of filter paper with 1 or 2 drops of lead acetate solution and hold it in the mouth of one of the bottles of gas. Introduce a lighted splinter. The other bottle invert over water and shake.

Use a Kipp's generator to supply the gas as needed;

pass it through small quantities of the following substances:

1. Concentrated sulphuric acid. What result?
2. Chlorine water.
3. Bromine water.
4. Dilute solution of iodine in potassium iodide.
5. Nitric acid (1 part acid to 1 part water).

Collect the precipitates in 2, 3, 4, and 5 on a filter, wash and scrape together, then place on the end of a platinum wire, curved to a small loop, and hold in a Bunsen flame (?). What odor is emitted? What object is there in using concentrated sulphuric acid or moderately concentrated nitric acid for the preparation of hydrogen sulphide? Explain what is meant by an oxidizing agent, and compare these reactions with that used in preparing chlorine.

Action on Bases

Experiment 77 (HOOD): Take 5 c. c. of potassium hydroxide solution from the side table and saturate with hydrogen sulphide. What is the product? What is its effect on lead acetate solution? zinc sulphate solution? Place a drop from a glass rod on a silver coin. Result?

Action of Salts

Experiment 78 (HOOD): Through 5 c. c. of copper sulphate solution pass hydrogen sulphide until no more precipitate forms, then filter, and to the clean filtrate add a few drops of ammonia solution. With 5 c. c. of zinc sulphate solution proceed just as with the copper sulphate. What difference is there in the result? What is the cause of the difference?

Using the results of these three experiments with hydrogen sulphide as a basis, classify sulphides according

to their solubility. For the sulphides which belong to each class consult your text-book.

Compounds of Sulphur with Oxygen, and with Oxygen and Hydrogen

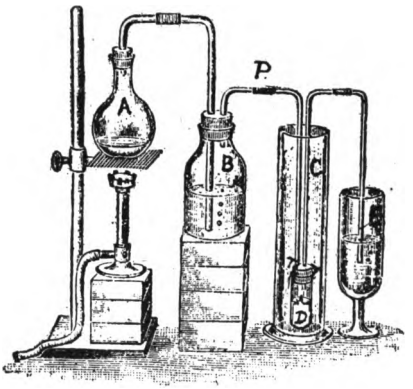

Fig. 12.—A, generating flask. B, wash-bottle of H_2SO_4. C, Cylinder containing freezing mixture. D, small t. t. E, beaker of water.

Sulphur Dioxide

Experiment 79: *a. Preparation* (Hood): Use an apparatus as shown in the cut. Place 20 g. of copper shavings in the generating flask, pour upon them 50 c. c. of concentrated sulphuric acid; heat. When the gas begins to pass off, so regulate the heat that the evolution of gas will not be too rapid. Disconnect at P and collect two small bottles of gas by displacement of air, and then, after again connecting at P, pass the remainder through the freezing mixture in C for twenty-five minutes; then remove D, after having first sealed it.

b. Properties (Hood): 1. Insert a burning splinter into one bottle; a strip each of moist red calico, yellow calico, and litmus paper into the second.

2. Neutralize a sulphur dioxide solution with potassium hydroxide solution and evaporate to dryness on the water-bath.

Manufacture of Sulphuric Acid

(See text under sulphuric acid.)

Experiment 80: The manufacture of H_2SO_4 may be prettily represented by the following process: A large glass globe or flask is filled with air or oxygen and provided with five tubes, as shown in figure.

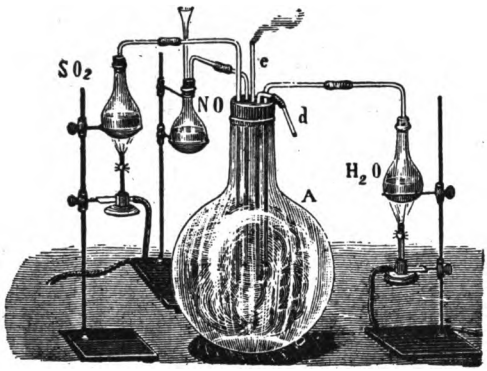

FIG. 13.

One tube connects it with a flask which furnishes a current of SO_2; another connects it with a second flask or bottle which furnishes a current of NO; the third connects it with a flask which furnishes a current of steam; by the tube d, a supply of air or O is admitted, from time to time, into the globe. The fifth tube, e, allows the escape of the waste products of the reaction; it may be connected with an aspirator.

a. NO enters the globe and takes O from the air. The ruddy fumes of NO_2 are seen.

b. On admitting a current of SO_2, the red fumes of NO_2 disappear and white "leaden-chamber crystals" form on the walls of the globe. The NO_2 has been reduced and the SO_2 oxidized.

c. On admitting steam, the crystals disappear, and dilute H_2SO_4 collects at the bottom of the globe.

d. If air be admitted, red fumes again appear and the process may be repeated.

Properties.—Into test-tubes, containing respectively a little cane-sugar and a small piece of wood, pour a few c. c. of concentrated sulphuric acid and allow to stand. Result?

CARBON

Carbon Dioxide

Experiment 81: Use a Kipp's generator, charged with marble and hydrochloric acid (1 to 2). (Why not use sulphuric acid?) Wash the gas through water, and, if dry gas is needed, through a second bottle containing concentrated sulphuric acid. Collect the gas by displacement of air. Why? Into one bottle filled with gas, insert a glowing pine splinter. Into another pour a little lime-water. Result? Pass carbon dioxide into 20 c. c. of a solution of calcium hydroxide until the precipitate which forms redissolves. Divide this solution into two parts; to one portion add hydrochloric acid, boil the other portion, then add hydrochloric acid. Result?

Carbonates

Experiment 82: Dissolve 4 g. of potassium hydroxide in 5 c. c. of water in a 100 c. c. Ehrlenmeyer flask. Fit it with a rubber stopper with one hole, through which passes a glass tube almost to the solution. Connect this tube with a carbon dioxide generator, loosen the stopper and replace

the air in the flask with this gas. Then stopper the flask tightly, and while the gas is still flowing into it shake it vigorously. When the absorption subsides, cool the flask by cold water, and continue the process until no more gas is taken up. Collect the solid product on a filter. One portion boil with water, the other dry between filter papers and heat in a tube. In both cases pass the escaping gas through lime-water. In the second case, heat until no more gas escapes. When the residue is cold, dissolve it in water and add a few drops of the solution to the solutions of each of the following salts: Barium chloride, calcium chloride, magnesium sulphate, copper sulphate. Filter in each case and test the solubility of the precipitate in acetic and in hydrochloric acid. What is disengaged by the action of the acid? (This is the test for carbonates.) Heat some secondary carbonate of sodium to redness in a crucible for half an hour. Is it still carbonate? Repeat with magnesium carbonate.

Percentage of Carbon Dioxide in Calcium Carbonate

Experiment 83: *a.* Place in the test-tube T enough H_2SO_4 to cover the end of K, then having placed about 30 c.c. of HCl in the thistle-tube H, whose neck is closed by the ground glass plug S, weigh carefully the entire apparatus. Now place about 1.5 g. of $CaCO_3$ in the flask F and weigh again. Be sure that all joints are perfectly tight, then carefully let in HCl from the thistle-tube, a few drops at a time, until all action ceases. Now connect D with an aspirator, and after all the CO_2 contained in F has been drawn out, weigh again and estimate the % of CO_2 in the $CaCO_3$ taken. Tabulate your results.

b. To determine the % of Ca in $CaCO_3$. Take another piece of weighed calcium carbonate, place in an evaporating-dish and add HCl of same strength as in *a*.

Evaporate to dryness. You now have all the calcium as calcium chloride. You know that the atomic weight of Cl is 35.45; then weigh and compute the amount of Ca in the substance. Find % of Ca in the calcium carbonate.

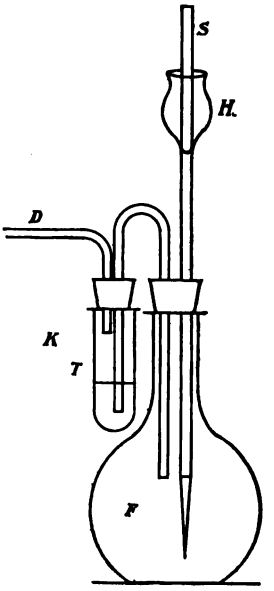

FIG. 14.

c. Upon adding the % of Ca to the % of CO_2, you will notice that the sum is less than 100 %. This third % is to be found in oxygen. Of course the % of O is 100 % — (% of CO_2 + % of Ca).

If each % is divided by the proper atomic weight of the element the quotients will be in the same ratio as the number of atoms of each element of the compound. CO_2 may be regarded as an element in this computation.

Divide the % of Ca by 40, the % of CO_2 by 44, and the % of O by 16, and you will have the simplest formula of the calcium carbonate. The molecular formula must be determined by other means, as the simplest formula is not always the molecular formula.

MAGNESIUM

To Determine the Weight of Acid required to Dissolve a Given Weight of Magnesium

Method explained.

1. A known weight of acid is neutralized by NaOH solution, and volume of NaOH noted.

2. The same weight of acid is again taken and a piece

of Mg placed in it. After the Mg is all dissolved, NaOH is added to this solution until neutralized. Volume noted.

3. The volume of NaOH in 1 is chemically equivalent to the weight of acid, and the volume NaOH in 2 is chemically equivalent to the weight of acid *not used* to dissolve the Mg. Hence,

4. Vol. 1 — Vol. 2 = vol. used in dissolving Mg. Finally, volume NaOH used to neutralize that acid which dissolved the Mg ÷ volume NaOH used to neutralize *total* acid *is* the *fractional part* of acid that dissolves Mg.

Experiment 84:

a. Chemicals: A piece of magnesium whose weight is accurately known; a solution of sulphuric acid containing exactly 25 grams of acid per liter of solution; an alkali of any convenient strength.

b. Operations: 1. Neutralize 10 c. c. of the acid solution with alkali, observing the exact volumes used. (Call this amount x. Use Phenolphthalein as an indicator.)

2. Dissolve the magnesium in 10 c. c. of the acid solution and neutralize the solution with alkali; call the number of c. c. used y.

c. Computations: Answer the following questions in terms of x and y, and also in terms of the numbers just obtained:

1.	What volume of the alkali necessary to neutralize 10 c. c. of acid solution?		
2.	What volume of alkali necessary to neutralize the solution containing the magnesium?		
3.	What volume of alkali solution necessary to neutralize that acid which has united with the magnesium? . .		
4.	What part of the alkali used in *a* would have neutralized that acid which has united with the magnesium?		
5.	What weight of acid in 10 c. c. of the solution?		

6.	What part of the acid used in *b* has united with the magnesium? . .			
7.	What weight of acid has united with the magnesium?			
8.	What weight of magnesium was used?			
9.	What weight of acid will unite with this weight of magnesium? Determine by use of the equation and molecular weights			
10.	Difference between the theoretical amount and the amount found by experiment			
11.	Percentage error			

PROBLEMS

1. A solution of sulphuric acid contained 80 g. per liter. 25 c. c. of the solution were used in dissolving a piece of zinc. It was found that 68 c. c. of alkali would neutralize 25 c. c. of the acid, but after the Zn had been dissolved in the 25 c. c. of acid that 22 c. c. of alkali would neutralize the remaining acid. What was the weight of the metal?

2. A sulphuric acid solution contains 50 g. of acid per liter. Ten c. c. of the acid solution is neutralized by 40 c. c. of NaOH. A piece of iron weighing .0714 g. is then dissolved in 10 c. c. of the acid solution. The remaining solution is neutralized by 30 c. c. of the NaOH solution. (a) What is the weight of acid used? (b) What weight of acid will unite with the above weight of iron?

WORK AND ENERGY

When anything by reason of its position or motion is capable of doing work, it is said to possess *energy*. When energy is applied to overcoming resistance, we say *work* is done. Energy is of two classes; viz., *potential* and *kinetic*.

Potential Energy

Potential energy is energy of position. A lake at the top of a hill or a mountain possesses potential energy; for

should its water be released it would be capable of doing work by running down the slope. The work which it can do is measured by the mass of the water multiplied by the distance through which it acts.

Kinetic Energy

The capacity to do work possessed by a body in motion is called *kinetic energy;* and the measure of this is one-half the mass multiplied by the square of the velocity or $\frac{MV^2}{2}$. See Physics KE $\frac{WV^2}{2g}$.

Conversion of Potential into Kinetic Energy

If a portion of the potential energy possessed by a body is converted into kinetic energy by its assuming motion, then the sum of the capacity for work still remaining, and of kinetic energy already produced, is equal to the potential energy originally contained in the body.

If you recall the burning of H in air, you remember heat was evolved. This heat is capable of doing work, such as heating water to form steam to drive an engine. H and O when in contact possess a form of energy called *chemical energy,* which is akin to potential energy, but because of insufficient means we cannot resolve the chemical energy into factors, weight and distance. This energy, however, can be measured by the amount of work which given weights of the interacting bodies are capable of performing. This energy is usually manifest in the form of heat which is capable of measurement.

The energy required to decompose a body is equal to that given off in its formation; for exactly as much kinetic energy must be used in decomposing a given quantity of water into its constituents, O and H, as was given off in the formation of the same weight of the liquid. That is,

the amount of electricity that we use to decompose water could perform just as much work as could the heat given off in the forming of the water which was decomposed.

KINETIC THEORY OF GASES

(*Methods of determining Molecular and Atomic Weights*)

I. Molecular Hypotheses

(*Brief Statement to be Amplified from Carhart and from Torrey*)

1. Molecules of the same gas are all alike, and are separated by spaces that are very great compared with the molecules themselves. This depends on the fact that, when heated, the colors of the gases are independent of pressure. If the molecules were close together mutual action would ensue, influencing the molecules' color. This shows that the color depends on the nature of the molecules, and not on the spaces between them.

2. Molecules move in straight lines between mutual encounters.

3. All molecules of the same gas have equal masses, and the average kinetic energy is the same for all molecules of all gases at the same temperature.

II. Theory of Pressure for Gases

1. Let M represent the mass of a molecule of any gas, and let V represent its average velocity. Then MV represents its momentum. Now, since the pressure of a gas on the walls of the containing vessel results from the united impacts of all the molecules, we have P varies as MV, where P represents the pressure.

But the pressure depends also upon the continuous impacts, or the number of impacts per second, and these

depend directly upon the velocity of the individual molecule.

Hence P varies as number of impacts varies, or as V.

2. P varies with V. But where one quantity varies with two variable quantities it varies according to their product.

Hence we have

3. P varies with MV^2.

We note further that the pressure varies also with the number of molecules in unit volume in the containing vessel, so that

4. P varies with N, where N represents the number of molecules.

Hence, as above,

5. P varies with NMV^2. These factors represent all the variable quantities with which, for gases, we are familiar. Therefore, to change our last expression in variation to an expression of equality (equation), we need simply to introduce a constant factor, as K, into one member. We have, then,

6. $P = KNMV^2$ for the pressure of a gas.

III. Avogadro's Law

For two gases at the same temperature, we have from the preceding topic, for unit volume,

1. $P_1 = KN_1M_1V^2_1$ and $P_2 = KN_2M_2V^2_2$.

If the pressures are equal, then

$$KN_1M_1V^2_1 = KN_2M_2V^2_2 \text{ or}$$

2. $N_1M_1V^2_1 = N_2M_2V^2_2$.

Now the gases are at the same temperature, and the temperature depends upon the kinetic energies of the molecules. Therefore, from the third hypothesis, the

kinetic energy of a molecule of one gas moving at the average velocity for that gas and the temperature must equal the kinetic energy of a molecule of the other gas, moving at the average velocity for the second gas at that temperature; i. e., $\frac{1}{2}M_2V^2_2$ or

3. $M_1V^2_1 = M_2V^2_2$. Dividing each member of the equation (2) by the corresponding member of equation 3, we get $\frac{N_1M_1V^2_1}{M_1V^2_1} = \frac{N_2M_2V_2}{M_2V^2_2}$. Canceling common factors (see 3) we get (4) $N_1 = N_2$. State the law. Corollary. Let D_1 and D_2 represent the densities of the above gases. Then (5) $D_1 = M_1N_1$ and $D_2 = M_2N_2$. But $N_1 = N_2$. Hence by dividing $D_1 : D_2 :: M_1 : M_2$, or the densities of the gases are proportional to their molecular weights.

IV. To Prove Number of Atoms per Molecule in Elementary Gases

1. Analysis determines the volumetric composition of HCl to be 1 vol. of H + 1 vol. of Cl = 2 vols. of HCl.

Also for ammonia:
1 vol. of N + 3 vols. of H = 2 vols. of NH_3.

And for water vapor:
2 vols. of H + 1 vol. of O = 2 vols. of H_2O.

2. Avogadro's Law applied to the *volumetric* composition of such gases as HCl, NH_3, etc., enables us to determine the number of atoms per molecule of elementary gases, for

(a) Equal volumes of H and Cl contain equal numbers of molecules, say x.

(b) Then, the volume of HCl resulting, which is twice the volume of H or Cl, contains $2x$ molecules.

(c) Hence, the x molecules of H spread over the $2x$

molecules of HCl, therefore each molecule of H breaks up into at least two parts, which means the molecule contains at least two atoms.

(d) The same reasoning can be extended to Cl, N, O, and to any gaseous element.

V. How the Molecular Weight of a Gaseous Compound is Determined

1. (a) Analysis by volume and the application of Avogadro's Law to the result, has proved that there is at least one atom of Cl joined to one atom of H to form the HCl acid molecule. We have no reason for assigning *more* than one atom of Cl and one of H to the molecule, and hence we shall assume that there is but one atom of each.

(b) In the next place, analysis *by weight* shows that one part *by weight* of H unites with 35.4 parts *by weight* of Cl. Hence, as we have shown that there is one atom of H and one of Cl in the hydrochloric acid molecule, its molecular weight could not be less than $1 + 35.4 = 36.4$. Otherwise we should have to assign to H an atomic weight less than unity.

2. Having adopted 36.4 as the molecular weight of hydrochloric acid, we may find the molecular weight of any gas whose specific gravity we can determine, for example:

Let D = the density of hydrochloric acid, and
Let M = the molecular weight of HCl.
Let D^1 = the density of any gas as sulphur dioxide and
Let M^1 = the molecular weight of sulphur dioxide.

Then, by Avogadro's Law.

$D : D^1 :: M : M^1$; substituting known values,

$18.2 : 32 :: 36.4 : M^1$. Hence $M^1 = 64$ = molecular weight of SO_2.

3. To prove that the molecular weight of a gaseous compound equals twice its density:

(a) First, determine the molecular weight of hydrogen by above method.

Let D = density of hydrochloric acid = 18.2.

Let M = mol. weight of hydrochloric acid = 36.4.

Let D^1 = density of hydrogen = 1.

Let M^1 = mol. weight hydrogen.

Then $D : D^1 :: M : M^1$ or $18.2 : 1 :: 36.4 : M^1$ therefore $M^1 = 2$ = molecular weight of hydrogen.

(b) Prove $M = 2D$.

Having determined that the molecular weight of hydrogen is 2, we may determine the molecular weight of any gas in the following manner:

Let D = density of hydrogen = 1 and M = its mol. weight = 2.

Let D^1 = density of any gas and M^1 = its mol. weight.

Then $D : D^1 :: M : M^1$, substituting known values, $1 : D^1 = 2 : M^1$ or $M^1 = 2D^1$.

4. If the density of a gas be referred to air = 1, then, the molecular weight of the gas = 28.88 times D; for the density of air referred to hydrogen is 14.44.

VI. General Method of Determining Atomic Weights

1. The weight of the hydrogen atom = 1 is the standard, and we use for reference also the atomic weights determined as above.

Chlorine = 35.4, nitrogen = 14, oxygen = 16.

2. We determine the *molecular weights of gaseous compounds* containing the given element, say, for example, carbon:

Name.	Sp. Gr.	Mol. Wt.
Carbon dioxide.....	22	44
Carbon monoxide.....	14	28
Methane	8	16
Cyanogen	26	52

3. A quantitative analysis shows that in

Forty-four parts by weight of carbon dioxide there are 32 parts by weight of O and 12 parts by weight of C; and in

Twenty-eight parts by weight of carbon monoxide there are 16 parts by weight of O and 12 parts by weight of C; also in

Sixteen parts by weight of methane there are 4 parts by weight of H and 12 parts by weight of C; and in

Fifty-two parts by weight of cyanogen there are 28 parts by weight of N and 24 parts by weight of C.

Hence an examination of all known compounds of carbon has shown that the smallest part by weight of carbon in any molecule is 12.

4. Give definition of atomic weight.

"Atomic weight is the lowest combining weight *per molecular* weight."

To Determine the Density of Chloroform Vapor

Experiment 85: *a. Definition.*—*Density* is the mass contained in unit volume. We have already learned that for solids and liquids the unit volume is one cubic centimeter, and the unit of mass is the gram. Now, for gases the unit of mass is the same, one gram, but the cubic centimeter is too small to be a convenient volume for measuring gases. Hence, because hydrogen is the lightest of gases, it has been agreed to assume as the unit volume for gases, the volume occupied by 1 gram of hydrogen at 760 mm.

and 0° C. Now, one liter of hydrogen at standard conditions weighs 0.0896 g., hence 1 g. of hydrogen occupies 1.0000/.0896 = approximately 11.2 liters.

Hence the practical definition is: Density is represented by the number of grams in 11.2 liters of the gas.

b. Apparatus.—Boiler, steam-jacket, vaporizing-vessel, rubber tube for connecting vaporizing-vessel, measuring-tube and rubber tube connecting it with the leveling-vessel, Victor Meyer vial, thermometer.

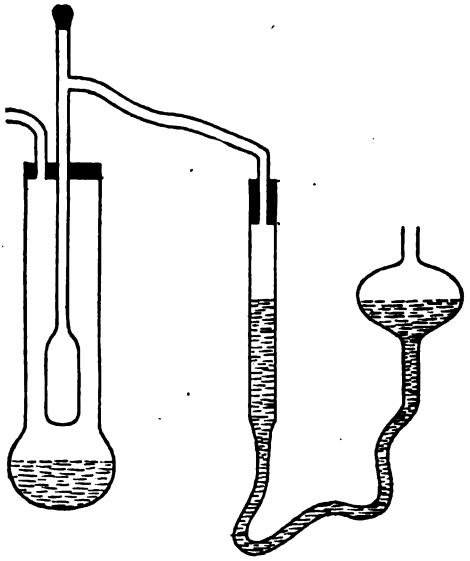

FIG. 15.

c. Operations.—Fill the leveling-vessel with water and raise it until the measuring-tube is filled nearly to the top of the graduated portion. Next suspend the vaporizing-vessel in the steam-jacket, connect by the rubber tube with the measuring-tube, and place on the upper end of the

vaporizing-vessel a rubber provided with two strong pinch-cocks. Loosen or remove the pinch-cocks until the vaporizing-vessel has come to the temperature of the steam in the steam-jacket. Then place the pinch-cocks on the rubber tubes and take the reading in the measuring-tube. Now fill the weighed vial with chloroform, and weigh again. Adjust the leveling-vessel until the level is the same in it and in the measuring-tube. Now place the filled vial in the rubber tube at the top of the vaporizing-vessel and between the two pinch-cocks. Next open the lower pinch-cock, permitting the vial to drop into the vaporizing-vessel. As chloroform boils at 61° C., it will readily evaporate in the vaporizing-vessel, driving air over into the measuring-tube. Lower the leveling-vessel, keeping it just below the level of the water in the measuring-tube until the level of the water becomes constant. Then equalize the levels again and take the second reading in the measuring-tube. What does this measured volume represent? Note the temperature of water in the leveling-vessel or tube; this will be the temperature of the gas measured. Take the barometer reading, temperature correction for barometer aqueous tension at the temperature of the gas. Record as follows:

First burette reading	c. c.
Second burette reading	c. c.
Volume of gas	c. c. observed.
Wt. of empty vial	g.
Wt. of vial and chloroform	g.
Wt. of chloroform	g.
Barometer reading	mm.
Temperature correction	mm.
Aqueous tension	mm.
Corrected pressure	mm. P.
Temperature of gas	t° Centigrade.

d. Computations.

1. Find the corrected volume of the gas from the formula.

$$V : V_0 :: \frac{P_0 : P}{T : T_0} \text{ or } V_0 = \frac{VPT_0}{P_0T}; \text{ find } V_0$$

NOTE 1.—The aqueous tension and temperature correction are both subtracted from the barometer reading to get corrected pressure P.

NOTE 2.—In the above formula

V represents observed volume.

V_0 represents corrected volume (to be computed).

P_0 represents standard pressure 760 mm.

P represents corrected pressure.

T represents observed temperature change to absolute scale (i. e., t° C. + 273° absolute temperature).

T_0 represents standard absolute temperature 273° C.

2. Knowing the weight of the chloroform and the corrected volume, V_0, compute the weight of 11,200 c. c. This will be the density; see definition.

3. From molecular weight = 2 × density, compute the molecular weight of chloroform, and from the formula $CHCl_3$ compare the result with the theoretical molecular weight.

4. Percentage of error? Difference between your result and true one? True result?

PROBLEMS

Vapor Density

1. At 750.538 mm. and 17° C., a volume of carbon dioxide measures 110 c. c. If the tension of aqueous vapor is 14.421 mm., barometer correction 2 mm., find the

corrected volume for standard conditions (e. g., 0° C. and 760 mm.).

NOTE 1.—The aqueous tension and barometer correction must *both* be subtracted from the observed pressure to get the corrected pressure.

NOTE 2.—Let V = observed volume; V_0 = corrected volume (X); P_0 = standard pressure (760 mm.); P = corrected atmospheric pressure; T = observed temperature changed to absolute scale (i. e., $t° + 273°$), where $t°$ = centigrade temperature; T = standard absolute temperature = 273° C.; then

$$V : V_0 :: \frac{P_0 : P}{T : T_0}, \text{ or } V_0 = \frac{VPT_0}{P_0T}; \text{ find } V_0.$$

2. A Victor Meyer vial contains .1035 g. liquid. The gas obtained measures 58 c. c. Barometer reading is 750 mm. Temp. correction, 2.5 mm. Temperature of gas, 27° C. Aqueous tension, 27.5 mm. (a) Find corrected volume. (b) Find the weight of one liter of the gas. (c) Find its specific gravity and molecular weight.

3. The following readings were obtained from a Victor Meyer experiment: Weight of liquid, .333 g.; volume of gas, 116 c. c.; barometer reading, 750 mm.; temperature correction, 2.5 mm.; temperature of gas, 27° C.; aqueous tension, 27.5 mm. Find corrected volume, weight of one liter, density, molecular weight.

4. What volume of chlorine may be prepared by using 19.401 g. of manganese dioxide?

5. What weight of limestone must be used to prepare 100 liters of carbon dioxide?

6. What volume of hydrogen sulphide may be produced from 88 g. ferrous sulphide?

7. Find the weight of 1 liter of hydrochloric acid gas.

8. How many g. in 10 liters of ammonia?

9. What is the weight of 100 liters SO_2?

10. How much will 5 liters NO weigh?

11. How many liters will there be in 19.8 g. of nitrous oxide?

12. How much will 10 liters of carbon dioxide weigh?

13. What weight $CaCO_3$ will be necessary to prepare the above weight (for 10 liters) CO_2?

14. What is the weight of 10 liters of carbon monoxide? What weight of oxalic acid is necessary to prepare it?

Determination of Volumes of Gases

Principle: The ratio between the numbers of molecules in two gases under the same temperature and pressure equals the ratio between the volumes of the same gases.

Hence, in solving problems for gases and volumes only, *first*, write the chemical equation; *second*, state a proportion in which the numbers of molecules of the two gases are proportional to the volumes.

1. Five liters of H burn in air. What volume of steam is produced? How much oxygen used? How much air?

2. How many cu. ft. of air will burn 8 cu. ft. of methane gas (CH_4)?

3. How many cu. in. of H will combine with 1 cu. in. of N? What is produced? How much?

4. What volume of oxygen is necessary to make 10 liters of carbon dioxide?

5. What volume of air is necessary to burn 25 cu. ft. of C_2H_6? What products are formed? Volumes?

6. Thirty liters of carbon disulphide burn in air. What is formed? What volume of each product?

7. How many c. c. H_2S will burn in 150 c. c. of oxygen? What volume is formed of each product?

8. Kerosene vapor is C_9H_{20}. Find the volume of air and of each product in burning 12 liters of the vapor.

9. A given sample of illuminating gas is 40 % H, 40 % methane, CH_4, 15 % carbon monoxide, CO, 3 % ethane, C_2H_6, and 2 % acetylene, C_2H_2. Find the volume of air necessary and the volume of gaseous products found in burning 100 cu. ft. of the gas.

10. Supposing gasoline to be pentane, C_5H_{12}, what volume of air would be mixed with 3 cu. ft. of its vapor to form the best explosive mixture for a gasoline engine?

Weights, Volumes, and Correction to Standard Conditions

1. It is necessary to use 6,250 c. c. of nitric oxide at 720 mm. and 27° C. What weight of nitric acid must be used to prepare it?

2. At 740 mm. and 27° C., 1,000 litres of sulphur dioxide were made from sulphuric acid and copper. What weight of acid was reduced?

3. At 750 mm. and 27° C., 7,250 c. c. carbon dioxide were obtained from limestone. What weight of limestone was used?

4. What volume of chlorine gas, at standard conditions, may be obtained from 585 g. of sodium chloride?

5. If 176 g. of ferrous sulphide are treated with hydrochloric acid, what volume of hydrogen sulphide at standard conditions will be formed?

6. If 25 liters of methane (CH_4) burn in air, what volume of air will be used? What volume of each product will be formed?

7. What volume of hydrogen will be necessary to unite with 18 liters of nitrogen? What volume of the product will be formed?

8. What volume of oxygen will unite with 20 liters of nitric oxide? What volume of the product will be formed?

9. What weight of limestone must be used to prepare 100 liters of carbon dioxide?

10. What volume will the gas in Problem 9 occupy at 27° C. and 730 mm.?

11. It is desired to use 50 liters of sulphuric dioxide gas at 17° C. and 740 mm. pressure. What weight of sulphur will it be necessary to burn in order to prepare the above amount of gas?

12. What other method could be used to prepare the above volume of sulphur dioxide? What weight of each?

Density, Hydrogen Standard

No.	Wt. of 1 liter of gas.	Density.	Mol. wt.	Percentage composition.	Molecular formula.	Name of comp.
1.	2.073 g.			C = 52.2 % H = 13 % O = 34 %		
2.	2.834			O = 76.2 % N = 22.2 % H = 1.6 %		
3.	3.3152			C = 64.9 % O = 21.6 % H = 13.5 %		
4.	4.1216			C = 39.1 % O = 52.2 % H = 8.7 %		
5.	1.6307			H = 2.7 % Cl = 97.5 %		
6.	0.7617			H = 17.6 % N = 82.4 %		
7.	2.8672			S = 50 % O = 50 %		
8.	3.4048			N = 36 8 % O = 63.2 %		
9.	2.43			H = 3.7 % N = 51.9 % C = 44.4 %		

Density, Air Standard

No.	Wt. of 1 volume air.	Wt. of equal vol. of gas.	Sp. gr.	M. W.	Percentage composition.	Molecular formula.	Name of comp.
1.	1,000 g.	1269.			H = 2.7 % Cl = 97.3 %		
2.		591			H = 17.6 % H = 82.4 %		
3.		2230			S = 50 % O = 50 %		
4.		2640			N = 36.8 % O = 63.2 %		
5.		939			H = 3.7 % N = 51.9 % C = 44.4 %		
6.		1530			N = 63.6 % O = 36.4 %		
7.		973			C = 42.8 % O = 57.2 %		
8.		1043			N = 46.7 % O = 53.3 %		
9.		1531			C = 27.2 % O = 72.8 %		
10.		973			C = 85.7 % H = 14.3 %		
11.		1042			C = 80 % H = 20 %		
12.		556			C = 75 % H = 25 %		

Determination of the Equivalent Weights of Magnesium and Iron

Experiment 86: *a. Definition.*—The equivalent weight of an element is the weight that will unite with or displace one part by weight of hydrogen.

b. Materials and Apparatus.—Magnesium ribbon, test-tube, evaporating-dish, burette, iron wire.

c. Operations.

A piece of magnesium ribbon 100 cm. long and weigh-

ing —— g. (determine this weight) is cut into pieces exactly 8 cm. in length to save time in weighing.

One piece is used for the experiment. Weight —— g.

A large test-tube is filled with water and inverted in an evaporating-dish of water. The magnesium ribbon is coiled around a small rubber band and placed under the inverted test-tube, so that the tube rests upon the ends of the rubber band in the bottom of the dish.

Next pour some sulphuric acid into the evaporating-dish. What reaction occurs? When the magnesium has all dissolved, hold the whole apparatus under the water-tap and allow the water to flow over it until tube and contents have reached the temperature of the water. Immediately mark with a small gummed label the depth of gas (what gas?) in the tube. Determine the volume of gas by admitting water from a burette.

d. Correction of Volume and Computation.

1. Correct volumes as in previous experiments. $V : V_0 :: P_0 : P :: T : T$ when V = observed volume.

V_0 = volume at standard conditions (to be computed).
P_0 = standard pressure = 760 mm.
P = observed pressure.
T = temperature of water (absolute scale).
T_0 = standard temperature, 0° C. ÷ 273 absolute.

2. Compute the weight of the corrected volume of hydrogen.
3. What weight of the magnesium displaces this weight of hydrogen?
4. Compute the weight of magnesium that would displace 1 g. of hydrogen. Conclusion.

Tabular Form

Metal.	Weights of metal.	Observ'd volume of H.	Correct'd volume of H.	Weights of H.	Equivalent wt. of metal.	Comput'd atomic weight.	True atomic weight.
Mg							
Fe							

PROBLEMS

Equivalent Weights

1. A piece of zinc weighing 0.0993 g. dissolves in sulphuric acid. The hydrogen evolved measures 38.8 c. c. at 742 mm. atmospheric pressure and 15° C. The temperature correction for the barometer is 2 mm., aqueous tension 12.8 mm. Find the volume of hydrogen at 760 mm. and 0° C., the weight of the hydrogen, and the equivalent of the zinc.

2. If 0.085 g. magnesium displaces 88.8 c. c. of hydrogen at 742 mm. and 15° C., temperature correction 2 mm., aqueous tension 12.8 mm., find the combining weight of the magnesium.

3. A few pieces of iron weighing .1008 g. dissolves in HCl. The hydrogen evolved measures 46.4 c. c. at 739 mm. and 27° C., temperature correction for the barometer is 2 mm., aqueous tension 17 mm. Find the combining weight for the iron.

4. A piece of zinc weighing 0.0731 g. dissolves in sulphuric acid, displacing 29 c. c. of hydrogen. If the pressure is 739 mm., temperature 27° C., barometer correction 2 mm., aqueous tension 17 mm., find the volume

of hydrogen at standard conditions, the weight of hydrogen, and the combining weight of zinc.

5. A strip of magnesium ribbon weighing 3 g. burns in air, and the resulting oxide weighs 5 g. If the equivalent of oxygen is 8, what does this determination give for the equivalent of magnesium?

6. If 24.3 g. of zinc oxide yield, upon reduction, 19.5 g. zinc, what is the equivalent of the zinc?

7. A piece of zinc weighing 0.975 g. dissolves in silver nitrate. The silver deposited weighs 3.24 g. If the equivalent of zinc is 32.5, what does the determination give for the equivalent of silver?

8. A piece of iron weighing 8 g. dissolves in copper sulphate. The copper deposited weighs 9 g. If the equivalent of iron is 28, find the equivalent of copper.

Equivalent Weight of Silver Displaced by Zinc

Experiment 87: Weigh out about .6 of a gram of clean zinc foil, place it in a small beaker and pour a concentrated solution of silver nitrate upon it, and allow it to stand. As the silver is gradually deposited, the zinc is as gradually dissolved, until finally it has entirely disappeared. When the zinc is completely gone, the liquid must be carefully decanted off, and the precipitated silver washed once or twice by half filling the beaker with water, stirring gently, allowing it to settle and again decanting the liquid. All the silver is next transferred to a clean porcelain crucible, which, with its lid, has been counterpoised carefully. When the whole has in this way been got into the crucible, the water is decanted and the silver drained as dry as possible. The crucible, partly covered with its lid, is then placed in a hot-air oven, heated to about 110° to 120°, in order to dry the silver perfectly; after which it is allowed to cool, and is then weighed. It

should then be returned to the oven for a second heating, and once more weighed. If this last weighing agrees with the one before, it shows that the substance was perfectly dry.

Example: Weight of zinc used = 0.48 gram.
Weight of silver obtained = 1.60 grams.
Then as $0.48 : 32.5 :: 1.6 : x$.

$x = \frac{32.5 \times 1.6}{0.48} = 108.3$ = Weight of silver displaced by 32.5 parts by weight of zinc.

By this experiment, then, 108.3 parts by weight of silver are equivalent to 32.5 parts by weight of zinc. But this weight of zinc is equivalent to 1 part by weight of

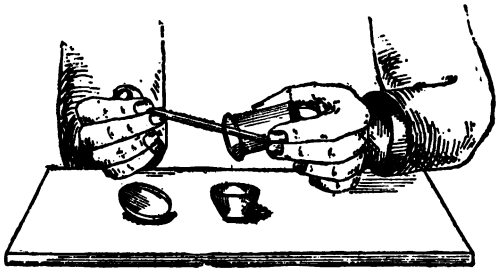

FIG. 16.

hydrogen; therefore we say that 108.3 parts by weight of silver are equivalent to 1 part by weight of hydrogen.

To Find the Weight of a Liter of Oxygen

Experiment 88: *a. Apparatus.*—A test-tube containing $KClO_3$ and MnO_2, a large bottle full of water and a smaller one to catch the water as it is forced out of the larger one when oxygen is generated in the test-tube. Weigh the

test-tube and attach it to the larger bottle by means of a bent tube, after having filled the delivery-tube with water and held it there by means of a pinch-cock.

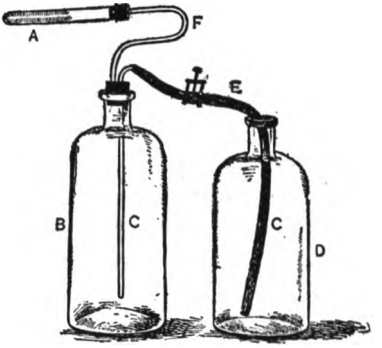

FIG. 17.

When ready to heat the test-tube, remove the pinch-cock, and as the oxygen enters the large bottle, water will fill the smaller one. When about three-quarters full, gradually decrease the heat and find the volume of water in the small bottle. This volume of water exactly equals the volume of oxygen. Weigh the test-tube again. Its loss of weight equals the weight of oxygen given off.

Computations.—Find the corrected volume of the gas from the formula.

$$V : V_0 :: \frac{P_0 : P}{T : T_0}, \text{ or } V_0 = \frac{VPT_0}{P_0T}; \text{ find } V_0.$$

NOTE 1.—The aqueous tension and the temperature correction are both subtracted from the barometer reading to get corrected pressure P.

NOTE 2.—In the above formula,

V represents observed volume.

V_0 represents corrected volume (to be computed).

P_0 represents standard pressure, 760 mm.

P represents corrected pressure.

T represents observed temperature changed to absolute scale (i. e., t° C. + 273 absolute temperature).

T_0 represents standard absolute temperature = 273° C.

You have now the weight of a given volume of oxygen

under the standard conditions of temperature and pressure. Find the weight of 1 liter of oxygen under similar conditions.

THE HALOGENS

Hydrofluoric Acid

Experiment 89 (HOOD): Coat the surface of a glass plate with a thin, even layer of paraffine, and with a sharp point carefully etch a design in the wax. Place one gram of calcium fluoride in a small leaden dish (provided) and pour on enough concentrated sulphuric acid to make a paste. DO NOT INHALE ANY OF THE FUMES GIVEN OFF. Cover the dish with the prepared glass. Allow to stand for an hour in the back of the hood. If by that time the glass is not dull where the paraffine has been scratched away, carefully warm the dish with a very small flame; then scrape the wax off the watch-glass; finally clean with a cloth moistened with turpentine.

Preparation of Chlorine

Experiment 90 (HOOD): Place 9 c. c. of water and 1 c. c. of concentrated hydrochloric acid into 20 cm. test-tube, and cool to temperature of hydrant-water. Add a *very little* lead dioxide from the corner of a glass spatula, adding slowly until the merest trace remains undissolved. Change of color in liquid? Odor? Now boil the solution, holding moist blue litmus paper over the mouth of the tube. Why? Odor? When the solution is nearly colorless, set aside to cool. Explain the change that takes place at the two temperatures. (See *Experiment* 49.)

Of the chlorine water prepared in Experiment 49 shake up 10 c. c. with a few copper filings, and then warm. Note the color of the solution. Why? Pour the remainder of the chlorine water into the bottle on the side-table.

In Experiment 89, what has happened to the glass? (To empty the leaden dish, place it in a large evaporating-dish, and quickly pour in a large amount of water; the liquid can then be safely poured down the waste-pipe.) What is the difference between the naturally occurring fluorides and the salts of the other halogens which are found as minerals?

Bromine

Experiment 91 (Hood): *a. Preparation.*—Measure 6 c. c. of water into a small Florence flask and to it add 24 c. c. of concentrated sulphuric acid. Cool well. Thoroughly pulverize in a mortar 15 g. of potassium bromide and mix it well on a piece of paper with an equal weight of powdered manganese dioxide. Pour this into the distilling-flask under the hood. Add the H_2SO_4, and allow it to stand for five minutes. Then shake well, and distil slowly into a small Florence flask; discontinue as soon as water begins to come over.

b. Properties (Hood).—Put a few drops of bromine into each of three bottles, cover them, and shake well. Why? Repeat *a*, *b*, and *e* under Experiment 50, using Br in place of Cl. Prepare the water solution by adding to 20 c. c. of water as much bromine as it will dissolve, and, in addition, try the action of bromine water on iron-filings. Why? To one c. c. of carbon bisulphide add one c. c. of bromine water, and shake. Why? Color of carbon bisulphide solution?

Iodine

Experiment 92 (Hood): *a.* Preparation.—Pulverize 4 g. of potassium iodide and mix thoroughly with an equal weight of manganese dioxide. Place the mixture in a middle-sized evaporating-dish embedded in a sand-bath,

and pour upon it a cold solution of 5 c. c. of concentrated sulphuric acid with 2 c. c. of water. Mix quickly and thoroughly with a glass rod, and after standing two minutes, pour in 10 c. c. of concentrated sulphuric acid; cover the dish afterward with a clean, dry watch-glass, partially filled with cold water. Warm so as to sublime the iodine with as little loss as possible. Why?

b. Properties.—1. Examine some of the most perfect crystals under a microscope.

2. Try their solubility in water; in carbon bisulphide; in alcohol; in ether; in chloroform; in an aqueous solution of potassium iodide. Use only a few drops of each solvent; note the color of the solution at first, and the change in shade as more iodine passes into solution.

3. To a piece of starch as large as a pea add 2 c. c. of water, and boil; then pour the product into a beaker containing about 300 c. c. of cold hydrant-water, and add a drop of iodine solution by means of a clean glass rod. Stir, then pour away three-fourths of the liquid, and refill the beaker with water. Continue this process as long as there is a perceptible color. (This is a test for the presence of iodine.)

4. To a few crystals of iodine in water, add a very few iron-filings, and shake.

Preparation of Ammonium Iodide

Experiment 93: Upon 0.25 g. of pulverized I, placed in a porcelain capsule, pour enough strong ammonia water to cover it, and allow it to stand for 15 or 20 minutes. At the end of that time stir up the powder at the bottom of the liquid, and pour a quarter of the contents of the capsule upon each of four small filters. Wash the powder well with cold H_2O, and then remove the filters with their contents from their funnels. Pin the filters to pieces of

board and allow them to dry without heating. When the powder is dry, it may be exploded by brushing it with a feather or by jarring it with a blow upon the table. The powder is nitrogen iodide.

Hydrobromic Acid

Experiment 94 (HOOD) : *Properties.*—To a small piece of potassium bromide in a test-tube add two or three drops of concentrated sulphuric acid. Why? Hold a strip of moist blue litmus paper in the escaping gas. Why? Blow over the mouth of the tube. Why? Note color and odor. Why? What gases are given off? What object is there to prepare the acid in this way? Cause of the difference between this reaction and that used in preparing hydrochloric acid?

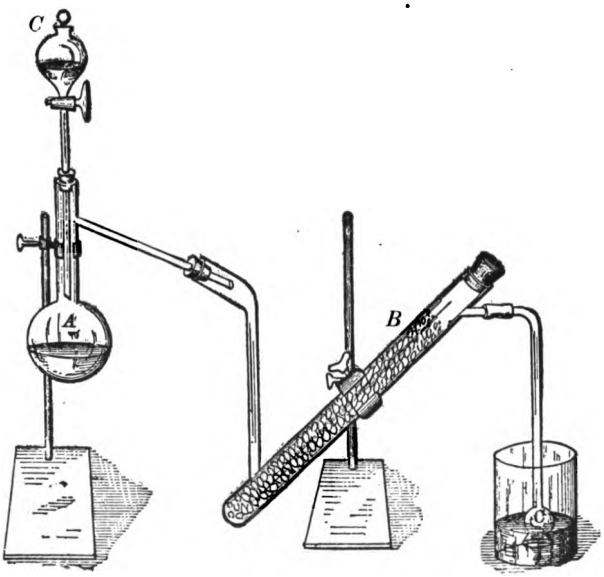

FIG. 18.

Experiment 95 (HOOD): *Laboratory method.* (Use apparatus in Fig. 18.) The flask A contains red phosphorus and barely water enough to cover the latter; the tube B is filled with small pieces of brick, covered with moist red phosphorus. The drop-funnel C contains bromine. Carefully open the stop-cock of the drop-funnel and let the bromine fall, drop by drop, upon the phosphorus cautiously. Fill two bottles with the escaping gas, and then conduct it into 10 c. c. of water until a drop of the solution on a piece of zinc causes a *decided* effervescence. The water must be placed in a beaker wide enough to receive the tube B, whose wide mouth should be just closed by the water. Why?

PHOSPHORUS

I. Preparation of Phosphine

(*Teacher's Experiments*)

CAUTION.—This gas is very poisonous, and must be prepared under the hood.

First Method

Experiment 96: Place three or four pieces of yellow phosphorus, each the size of a bean, in a flask containing about 200 c. c. of strong potassium hydroxide solution. Add 2 c. c. of ether. Fit the flask with a single-bored stopper and a one-bend delivery-tube, the delivery-tube dipping under water in a small jar. Arrange the flask over a sand-bath on the ring-stand and place the whole apparatus under the hood. Pull down the hood door after placing a low flame under the flask. Heat *very gently.*

The ether will be evaporated readily, driving the air out of the flask. After about ten minutes of gentle heating, chemical action will begin between the phosphorus

and the potassium hydroxide. This chemical action consists in the production of phosphine and (probably) potassium primary hypophosphite. KH_2PO_2; see hypophosphorus acid H_3PO_2.

At this stage the phosphine begins to escape from the flask and takes fire spontaneously on contact with the air. Question: Why was it necessary to place ether in the flask? It is known that pure phosphine is not spontaneously combustible, but that combustion in this case is started by the presence of very small amounts of the compound P_2H_4.

Equation for preparation of phosphine:

$$3KOH + 4P + 3H_2O = PH_3 + 3KH_2PO_2.$$

Second Method

Experiment 97: By the action of water or dilute acids on calcium phosphide, Ca_3P_2. Place a few small pieces of calcium phosphide in a beaker under the hood. Add a little water, and close the hood door. Phosphine will form rapidly, taking fire on coming in contact with the air.

Equation for preparation:

$$Ca_3P_2 + 6H_2O = 2PH_3 + 3Ca(OH)_2.$$

Compare formula of phosphine with formula of hydrochloric acid, hydrogen sulphide, ammonia. Which element does P resemble?

To investigate spontaneous combustion of phosphorus, burning phosphorus under water, etc., see Remsen.

Experiments with acids and salts of phosphorus I. (Topic for the note-book.) On burning in the air phosphorus forms phosphoric anhydride: $2P + 5O = P_2O_5$. This anhydride greedily absorbs water, forming, if a limited amount of water be used, *meta*phosphoric acid, a transparent, glossy solid.

$P_2O_5 + H_2O = 2HPO_3$. Compare with nitric acid,

$N_2O_5 + H_2O = 2HNO_3$. Upon treatment with excess of water, metaphosphoric acid takes up water to form orthophosphoric acid; the orthophosphoric acid appears as a white crystalline solid. This anhydrous orthophosphoric acid, at a red heat, separates off one molecule of water between two molecules of the acid and forms *pyro*phosphoric acid—$H_4P_2O_7$.

Equation: $2H_3PO_4 + \text{heat} = H_4P_2O_7 + H_2O$.

If pyrophosphoric acid be heated further, metaphosphoric acid is formed.

The above acids are readily neutralized with alkalis, the corresponding salts being formed.

a. Metaphosphoric acid forms metaphosphates. $HPO_3 + NaOH = H_2O + NaPO_3$, sodium metaphosphate. (Compare with action of KOH on HNO_3.)

b. Orthophosphoric acid forms orthophosphates. Three classes, primary, secondary, and normal, are formed.

1. $H_3PO_4 + NaOH = H_2O + NaH_2PO_4$, Sod. pri. orthophosphate.

2. $H_3PO_4 + 2NaOH = 2H_2O + Na_2HPO_4$, Sod. sec. orthophosphate, called also sodiumhydrogenphosphate. This is the common variety.

3. $H_3PO_4 + 3NaOH = 3H_2O + Na_3PO_4$, Sod. normal orthophosphate.

c. Pyrophosphoric acid forms two classes of pyrophosphates.

1. $H_4P_2O_7 + 2NaOH = Na_2H_2P_2O_7 + 2H_2O$.

2. $H_4P_2O_7 + 4NaOH = 4H_2O + Na_4P_2O_7$, Sod. nor. pyrophosphate.

II. Derivation and Tests for One Typical Salt of each of the Phosphoric Acids

Derivation of a Metaphosphate and Test

Laboratory Experiment 98: For a laboratory test, sodium metaphosphate can most conveniently be prepared by heating microcosmic salt. This compound is a salt of orthophosphoric acid, and is known otherwise as sodium ammonium hydrogen phosphate, $NaNH_4HPO_4$.

Place about 1 gram of microcosmic salt in a small test-tube, incline the mouth downward, and heat as long as any change continues. Note the odor of the escaping gas. What substances are driven off?

Equation: $NaNH_4HPO_4 + \text{heat} = NH_3 + H_2O +$ $NaPO_3$, sodium metaphosphate. What is the appearance of the residue in the test-tube?

When cool, dissolve in distilled water. Add one drop of silver nitrate solution. What forms at the surface? Color? What change occurs as this precipitate settles through the excess of sodium metaphosphate solution? The ppt. is silver metaphosphate. What can you say of its solubility in excess of sodium metaphosphate?
ver metaphosphate, white, soluble in excess of sodium

Equation: $NaPO_3 + AgNO_3 = NaNO_3 + AgPO_3$, sil- metaphosphate. (Note, if any yellow color appears, its source may be traced to insufficient heating of the microcosmic salt.)

Having tested the sodium metaphosphate satisfactorily, take a small portion of microcosmic salt, dissolve in distilled water, and add silver nitrate. What color? Note that microcosmic salt is an *ortho*phosphate, and this precipitate is a test for the phosphate.

Equation: $NaNH_4HPO_4 + 3AgNO_3 = HNO_3 +$ $NaNO_3 + NH_4NO_3 + Ag_3PO_4$, Sil. ortho. phos. *yellow.*

Test for an Orthophosphate

Experiment 99: As stated above, disodium hydrogen phosphate is the common orthophosphate. Dissolve a little disodium phosphate in some distilled water and add silver nitrate. Note color of ppt. This is the test for an orthophosphate.

Equation: $Na_2HPO_4 + 3AgNO_3 = 2NaNO_3 + HNO_3 + Ag_3PO_4$, silver normal orthophosphate, yellow. We have here a peculiar reaction: sodium secondary phosphate is neutral to indicators, silver nitrate is neutral to indicators, but upon mixing them we obtain products showing an *acid* reaction.

Test for a Pyrophosphate

Experiment 100: Place one gram of sodium secondary orthophosphate in a small test-tube and heat as long as a change continues.

Equation: $2Na_2HPO_4 + \text{heat} = H_2O + Na_4P_2O_7$, sodium pyrophosphate, amorphous white mass. When cool, dissolve in distilled water and add one drop of silver nitrate solution. Does the ppt. dissolve as it settles through the excess of sodium pyrophosphate solution? Compare with the action of silver nitrate in sodium *metaphosphate.*

Equation: $Na_4P_2O_7 + 4AgNO_3 = 4NaNO_3 + Ag_4P_2O_7$, silver pyrophosphate, white, insoluble in excess of the reagent.

Phosphorous acid. When phosphorus slowly oxidizes in moist air phosph*orous* anhydride is formed, $2P + 3O = P_2O_3$. This dissolves in water to form orthophosphorous acid. $P_2O_3 + 3H_2O = 2H_3PO_3$, orthophosphor*ous* acid.

The above acid forms salts similar to the salts of phosphor*ic* acid. Conclusion. We cannot fail to notice the similarity between the elements phosphorous and nitrogen.

They form hydrogen compounds of similar formula. Metaphosphoric and nitric acids are similar. They show further similarity in their anhydrides. Phosphorus forms a larger number of acids.

ARSENIC

Arsenic from Arsenous Oxide

Experiment 100: Prepare two "closed" (i. e., closed at one end only) tubes as follows:

Heat at its middle portion a common glass tube 20 cm. long. Use the luminous flame. Turn the tube slowly in the flame, heating a portion about 5 cm. long. As the tube softens draw it apart gradually, and finally seal off each of the tapering ends. Now place one-fourth g. of arsenous oxide in the closed tip of one tube. To transfer the arsenous oxide from the bottle to the tube, crease a piece of paper sharply and pour from the creased paper directly into the tube. Crowd a small piece of charcoal into the tube after the oxide, bringing the charcoal to within 2 cm. of the oxide. Next heat in the flame, holding the tube so that the charcoal is heated first nearly to redness. Next heat the portion of the tube containing the arsenous oxide, and notice that as it vaporizes it is forced to pass over the heated charcoal. Questions: What is the appearance (color, etc.) of the sublimate forming inside the tube above the charcoal?

What was the color of the original arsenous oxide? What element does heated charcoal (carbon) usually remove from an oxide with which it is in contact? What is reduction? oxidation?

Break the tube at the place where the band of sublimate has formed. What free element appears at this point? What is its color?

Equation: $As_2O_3 + 3C = ?$

Preparation of Arsine

(Teacher's Experiment)

Experiment 101: *CAUTION.—This gas is intensely poisonous, and must be prepared under the hood.*

Set up a hydrogen generator, providing the delivery-tube with a platinum tip. After testing the gas (H) until it gives a satisfactory result for pure hydrogen, light the jet, and observe that the hydrogen burns with an almost invisible flame. Hold a cool porcelain surface in the flame. If pure materials have been used, no deposit will form on the porcelain.

Next introduce through the safety-tube of the generator (the hydrogen jet still burning) one or two c. c. of a solution of arsenous oxide in dilute hydrochloric acid. Notice immediately any change in the appearance of the flame. Is any vapor apparently given off from the flame? Hold a cool porcelain surface in the flame, and observe any deposit. Compare this deposit with the deposit of free arsenic in the glass tube used in the preceding experiment. Next heat the glass tube about 5 cm. back from the burning-point and observe any deposit.

(NOTE.—This deposit may be driven along by the flame.)

Reactions.—The arsenous oxide on coming in contact with the nascent hydrogen in the generator is reduced, arsine, AsH_3, and water being formed. At the burning jet the arsine comes in contact with burning hydrogen and is decomposed, then burned, the hydrogen forming water and the arsenic burning to form arsenous oxide again.

Here explain the appearance of the white vapors near the flame. When the cool surface is held in the burning jet, the arsenic is cooled below the kindling temperature

for arsenic, free arsenic being thus deposited on the cool surface. The hydrogen continues to burn.

When the tube conveying the arsine is heated, arsine is decomposed into free arsenic and hydrogen. Hence, free arsenic is deposited inside the tube.

CAUTION.—Be sure to keep the hydrogen jet constantly burning. The arsine gas is intensely poisonous, and easily absorbed through breathing. After burning in the flame, solid arsenous oxide is formed. This, of course, is not readily inhaled, and any danger by inhaling is averted.

Equations: $Zn + H_2SO_4 = ZnSO_4 + H_2$.

For arsine: $As_2O_3 + 6H_2(\text{nascent}) = 2AsH_3 + 3H_2O$.

For burning arsine in free supply of air: $2AsH_3 + 3O_2 = As_2O_3 + 3H_2O$.

For burning arsine in contact with cool surface: $4AsH_3 + 3O_2 = 4As + 6H_2O$.

Anhydrides and Acids of Arsenic

Arsenic, like phosphorus, forms two anhydrides: As_2O_3 and As_2O_5.

The arsenic pentoxide with water forms orthoarsenic acid.

$$As_2O_5 + 3H_2O = 2H_3AsO_4. \quad (\text{Compare } HNO_3.)$$

If the solution of orthoarsenic acid be evaporated and the solid residue heated to 180°, then pyroarsenic acid is formed.

$$2H_3AsO_4 + \text{heat } (180°) = H_2O + H_4As_2O_7.$$

If further heated to 206°, meta-arsenic acid is formed.

$H_4As_2O_7 + \text{heat}\,(206°) = 2HAsO_3 + H_2O$. (Compare with nitric acid.)

Arsenous acid is known only in its salts. When liber-

ated from these it breaks down into the anhydride and water.

Comparing the hydrogen compounds, the oxides, and the acids of arsenic with the corresponding compounds of phosphorus, we see a close resemblance between the elements. They are classed in the same family.

ANTIMONY

Preparation of Antimony

Experiment 102: Place in the second of the closed tubes prepared for arsenic about one-quarter gram of antimonous oxide. Arrange above it a piece of charcoal as with the arsenous oxide, and heat as in that experiment. A higher temperature is required to vaporize the antimonous oxide; also the band of metallic antimony forms much nearer the charcoal, showing that antimony is not so volatile as the arsenic. Break the tube and examine the antimony. The antimony forms a whiter, more silver-like mirror than the arsenic.

Equation: $Sb_2O_3 + 3C + \text{high-red-heat} = ?$

Preparation of Stibine

(*Teacher's Experiment*)

Experiment 103 (Hood): Observing the same precautions as in the preparation of arsine, prepare a hydrogen generator, light the jet, and hold a cool surface in the flame. Noting the color of the hydrogen flame, and the lack of deposit on the cool surface, next add one or two c. c. of a solution of antimonous oxide in hydrochloric acid. In this solution, antimonyl chloride (SbOCl) is formed. As soon as the antimonyl chloride is added through the thistle-tube, observe any change in the appearance of the flame. Compare this with the arsine flame.

Next hold a cool porcelain surface in the flame. Compare the deposit with deposit of arsenic obtained from the arsine flame. Heat the glass tube, and note that the antimony mirror cannot be driven along. Compare with arsenic.

Reactions.—The antimonyl chloride in contact with nascent hydrogen is reduced to stibine, SbH_3. This, upon coming in contact with the burning hydrogen in the jet, is decomposed and the elements burned separately, water and antimonous oxide being formed. The porcelain surface when held in the burning jet cools the flame below the kindling temperature for antimony, and so the free element is deposited. Also in heating the glass tube, stibine is decomposed, antimony being deposited inside the tube. But antimony is too non-volatile to be further volatilized and driven along by heat, as the arsenic mirror can so easily be manipulated in this way.

Equation for forming hydrogen:

$$Zn + H_2SO_4 = ZnSO_4 + H_2.$$

For reducing antimonyl chloride:

$$2SbOCl + 3H_2(\text{nascent}) = 2SbH_3 + 2H_2O + 2HCl.$$

For burning stibine in free flame:

$$4SbH_3 + 6O_2 = 2Sb_2O_3 + 6H_2O.$$

For burning stibine against a cool surface:

$$4SbH_3 + 3O_2 = 4Sb + 6H_2O.$$

Anhydrides and Acids of Antimony

Like arsenic, antimony forms two anhydrides and only one of the corresponding acids. Antimonic anhydride (Sb_2O_5) dissolves sparingly in water to form orthoantimonic acid.

$$Sb_2O_5 + 3H_2O = 2H_3SbO_4. \quad (\text{Compare } HNO_3.)$$

Evaporation of this solution and heating the residue to 100° forms pyroantimonic acid. $2H_3SbO_4 + \text{heat}\ (100°) = H_2O + H_4Sb_2O_7$, pyroantimonic acid. Further heating of pyroantimonic acid to 200° forms meta-antimonous acid.

$$H_4Sb_2O_7 + \text{heat}\ (200°) = 2HSbO_3 + H_2O.$$

Prepare a summary of the study of nitrogen, phosphorus, arsenic, antimony, and place in the note-book.

SILICON

In its various compounds silicon is one of the most widely distributed elements on the earth's surface. As a free element it does not occur in the native state. Artificially, however, it may be prepared in the three following allotropic forms, resembling carbon in this respect:

1. If potassium fluosilicate (K_2SiF_6) be melted up with metallic aluminium, the fluorine unites with the aluminium and potassium, while silicon is obtained in the free state.

$4Al + 3K_2SiF_6(\text{fused}) = 4AlF_3 + 6KF +$ a part of the silicon in needle-shaped crystals, grayish-black. These crystals cannot be ignited in the air.

2. Melted iron and zinc can dissolve silicon.

$SiO_2 + C + Fe(\text{fused}) = CO_2 + Fe + Si$. Silicon is here in graphitoidal form and cannot be ignited in air.

3. Metallic sodium, heated, will decompose silicon chloride.

$SiCl_4 + 4Na\ (\text{heated}) = 4NaCl + Si$. This is in the form of an amorphous brown powder and easily burns in the air.

Silicon forms one compound with hydrogen, SiH_4, silicon hydride. This corresponds to the highest hydride of carbon, CH_4, methane.

Silicon Dioxide

Silicon unites with oxygen, forming silicon dioxide, SiO_2. In formula this compound resembles carbon dioxide, and, like carbon dioxide, it may be considered the anhydride of an acid, in this case meta-silicic acid.

$H_2SiO_3 - H_2O = SiO_2$. (Compare $H_2CO_3 - H_2O = CO_2$.)

As common sand, silicon dioxide occurs most widely distributed. The various hues in sand are caused by small portions of metallic oxide; most red colors are caused by the presence of ferric oxide, many greens by ferrous compounds. Quartz and rock crystal are crystalline forms of nearly pure silicon dioxide. In the amethyst we have the same substance colored by a slight trace of manganese. In agate the coloring matter is arranged in layers, giving the stratified appearance. In flint, jasper, chalcedony, we have further varieties of silicon dioxide differently colored. All forms of silicon dioxide show the conchoidal fracture.

Metasilicic Acid

Many salts of metasilicic acid are known. Sodium metasilicate (Na_2SiO_3) is one of the few soluble compounds of silicon. If this be treated with cold dilute hydrochloric acid, colloidal metasilicic acid is formed, $Na_2SiO_3 + 2HCl = 2NaCl + H_2SiO_3$. The sodium chloride may be separated from the metasilicic acid by dialysis. Upon heating, the acid breaks up into water and silicon dioxide.

$Na_2SiO_3 + 2HCl$ (hot concentrated) $= 2NaCl + H_2SiO_3$.

$H_2SiO_3 +$ heat $= H_2O + SiO_2$. Pure silicon dioxide is pure white in color.

Several important salts of metasilicic acid occur in nature. Calcium silicate ($CaSiO_3$) occurs as the mineral

wollastonite. Hornblende is a more complicated silicate of calcium, while asbestos is a variety of hornblende; its composition is approximately $CaMg(SiO_3)_2$.

When colored green by chromic oxide, the crystalline metasilicate of glucinum and aluminium is called emerald, $Gl_3Al_2(SiO_3)_6$.

Of the artificial silicates, window-glass, $Na_2Ca(SiO_3)_2$, and Bohemian glass, $K_2Ca(SiO_3)_2$, are the most common.

Orthosilicic Acid

Chiefly through its salts orthosilicic acid is of importance. It is interesting to note that in its composition it may be considered as derived from metasilicic acid by the addition of water to the latter. In this respect its formation is similar to the formation of orthophosphoric acid and arsenic acid by the addition of water to their respective meta acids. $H_2SiO_3 + H_2O = H_4SiO_4$, orthosilicic acid. This reaction probably takes place in the solution in which metasilicic acid is formed. Common clay is the aluminium salt of orthosilicic acid, $AlHSiO_4$. Its formula is also given $Al_2(HSiO_4)_2$. Other important orthosilicates are olivin, $Mg_2SiO_4Fe_2SiO_4$; garnet, $Ca_3Fe_2SiO_4)_3$; mica, a complicated orthosilicate.

Trisilicic Acid

By the separation of water from three formula weights of orthosilicic acid, trisilicic acid is formed, $3H_4SiO_4 - 4H_2O = H_4Si_3O_8$. The most important mineral derived from this acid is feldspar, potassium aluminium trisilicate, $KAlSi_3O_8$. This has a pink or flesh color, is very soft, and has cleavage in two planes. Feldspar is of special importance, in that it is a chief constituent of granite rocks. Granite, upon weathering and disintegrating, forms the clay soils which are of so much importance in

agriculture. A number of other silicic acids of less importance are known. In summing up, we may note four principal classes of silicon compounds:

1. Minerals consisting of silicon dioxide, quartz, rock crystal, agate, amethyst, chalcedony, onyx, flint, jasper.

2. Metasilicates. Hornblende, asbestos, glass, emerald, etc.

3. Orthosilicates. Clay, olivin, garnet, mica.

4. Trisilicates. Feldspar. The feldspars form an important group of minerals. The one of the formula $KAl(Si_3O_8)$ is most common.

THE PERIODIC SYSTEM

Up to this point in our work we have studied the following elements, together with several compounds of each: Hydrogen, oxygen, fluorine, chlorine, bromine, iodine, sulphur, selenium, tellurium, nitrogen, phosphorus, arsenic, antimony, carbon, silicon, calcium, magnesium, aluminium, zinc, copper, sodium, potassium.

If we place them by families of elements we have the following arrangement:

Li	Br	B	C	N	O	F
Na	Mg	Al	Si	P	S	Cl
K	Zn			As	Se	Br
Cu				Sb	Te	I

As a further classification of the elements and their properties, we wish now to arrange them in the order of their atomic weights. In this arrangement (omitting hydrogen and helium), we write the symbol for lithium, Li (an alkali metal similar to sodium); glucinum, Gl (a metal similar to magnesium); Boron, B (similar to aluminium); carbon, C; nitrogen, N; oxygen, O; fluorin, F. In this series of seven elements (omitting hydrogen

and helium), Li, Gl, B, C, N, O, F, we pass by regular gradations from the strongly metallic elements, lithium and glucinum, to the strongly non-metallic, negative elements, oxygen and fluorine.

Notice that the highest atomic weight reached is 19 for fluorine, which is the most negative element in the series. Now our next atomic weight in this increasing order is 23 for sodium. Sodium, as we know, is not similar to fluorine, but does resemble lithium. In our arrangement, then, we let sodium begin a new series, writing its symbol beneath that of lithium. In a similar manner and for similar reasons we place magnesium (Mg) beneath glucinum; Al beneath boron; Si beneath carbon; P under nitrogen, and so on. Continuing this system for the twenty-one elements lowest in atomic weight, we have the periodic arrangement. (See text, p. 313.)

We notice at once a striking similarity between this table and the table of arrangement by families as shown above. So far as the arrangement by families was carried out in part, this latter arrangement in order of atomic weights is precisely identical with the first arrangement. The three elements following manganese—i. e., iron, cobalt, and nickel—are very much alike and do not belong to any one of the main groups. We place them in a group (the eighth) by themselves. This eighth group is sometimes called a transition period between the third and fourth periods. The next element in order is copper. It has some properties which ally it to Group I. The following six elements fall in their proper places in Groups II to VII, and the following six elements fall regularly in Groups I to VI, so far as their properties are concerned. After molybdenum, in the sixth series, there is a blank. No element is known for this place, but it is probable that there is an undiscovered element with properties similar

to manganese. The next three elements resemble each other as closely as do iron, cobalt, and nickel, while silver has properties which ally it to Group I. This principle of arrangement was discovered by the German chemist, Lothar Meyer, and independently by the Russian, Mendeleeff. We observe that with each series of the elements from left to right we have in each case a regular decrease of metallic and increase of non-metallic characteristics.

Placing the eighth element under the first (lithium), the fifteenth under the eighth, we notice that the natural families or groups fall in columns. The horizontal lines or series of seven each are called "periods." We notice that similar properties of the elements recur with each seventh element in order of atomic weights, and we call this apparent *recurring dependence* of the properties of elements upon their *atomic weights* a *Periodic Function* of the atomic weights.

Hence the *Periodic Law:*

Properties of elements are periodic functions of their atomic weights.

The arrangement itself is called the *Periodic System.*

We will notice now some general formulas that will apply to each member of a given family. Representing an atom of any element by the symbol "M," we have for the highest oxide in each group, M_2O, for Group I; MO, M_2O_3, MO_2, M_2O_5, MO_3, M_2O_7, for Groups II to VII. This shows that the highest valence toward oxygen for the elements in the seven groups increases regularly from 1 to 7 in passing from Group I to Group VII. Similarly the compounds with hydrogen are represented by the formulas MH_4, MH_3, MH_2, MH, in passing from Group IV to Group VII. A decreasing valence toward hydrogen occurs in passing from Group IV to Group VII. The

general formulas for chlorides are MCl for Group I, MCl_2 for Group II, MCl_3 for Group III, MCl_4 for Group IV.

(See text for arrangement of the elements up to iodine.)

INTRODUCTION TO THE STUDY OF THE METALLIC ELEMENTS

Referring to the chemical elements studied up to this point, it will be seen that a definite order has been followed.

I. The Halogen Group: chlorine, bromine, iodine, or from the very non-metallic gaseous element chlorine to the *less* non-metallic solid element iodine.

II. The Sulphur Group: sulphur, selenium, tellurium; from the non-metallic sulphur to the somewhat metallic tellurium.

III. The Phosphorus Group: from the non-metallic phosphorus to the metallic element antimony.

IV. Carbon and Silicon.

In other words, we commenced our work with a study of the most non-metallic elements, and have finally, in the elements antimony and silicon, reached the dividing line between metals and non-metals. In each group also we have seen a gradual change in properties from strictly non-metallic properties to properties bordering upon the metallic; note chlorine to iodine, phosphorus to antimony.

It will now be our purpose to study the metallic or base-forming elements; and it appears best to take up first those having the most strongly marked characteristics of metals. Those elements are sodium, potassium of Group I, magnesium and calcium of Group II, etc. There are, however, certain incidental properties of solubility and precipitation which enable us to detect the presence of metals by analysis.

For purposes of analysis the classification of metallic elements does not follow strictly the order by families shown in the periodic system. In our study of metals we shall, therefore, follow the best order for analysis, and not adhere strictly to the periodic system.

We shall take up sodium, potassium, lead, silver, mercury, in the order named. Follow with a study of copper, iron and aluminium, zinc, calcium, barium, and magnesium. (For study of sodium and potassium, see text.)

NOTE.—In the following experiments use only chemically pure (C.P.) reagents and distilled water.

LEAD

Occurrence. The most important source of lead in nature is lead sulphide, PbS, known as galena. Lead occurs also in combination as the sulphate, carbonate, and chromate.

Metallurgy. Definition: Metallurgy is the process of extracting a metal from its ores. Two methods are in use for the extraction of lead from its ores:

1. By heating lead sulphide with metallic iron, free lead and iron sulphide are obtained. $PbS + Fe = FeS + Pb$.

2. By roasting the sulphide it is partly changed to lead oxide and lead sulphate. Upon heating a mixture of the two compounds in a closed vessel with more lead sulphide, the whole of the lead is obtained as the free element.

Solubility. Nitric acid is the best solvent for lead. Lead nitrate, nitric oxide, and water are the products. The chemical equations for all reactions of this kind are based upon accurate quantitative experiments. An inspection, however, of the class of reactions represented by the solution of lead reveals the fact that each reaction takes place in accordance with a definite law of oxidation and

reduction. A clear understanding of this law is a great aid in chemical calculations. In its several clauses, therefore, we shall state it at this point of our work.

Laws of Oxidation and Reduction

1. *Measure.* Oxidation (or reduction) is measured in units of valence; the valence of hydrogen = 1. In compounds, H is the standard unit.

2. *Free elements.* The valence of a free element is zero.

3. *Hydrogen and oxygen.* In compounds, the valence of hydrogen is always + 1, of oxygen always — 2. The valence for other elements varies; for hydrogen and oxygen in compounds it is constant.

4. *Oxidation and reduction.* An increase in valence from a negative valence up to zero, or from zero to a positive unit of valence, is oxidation. Any decrease in valence is reduction.

5. *Relation between oxidation and reduction.* In any reaction, the total amount of oxidation in units of valence always equals the total reduction in units of valence. We base our equations upon this principle.

6. *Determination of the valence of an element in a given formula.* The sum of the valences of all the elements named in a formula is zero. Remembering that hydrogen is always + 1, and oxygen — 2, we subtract the total hydrogen valence (or its equivalent) from the number representing the total oxygen valence. The difference is the valence of the element in question.

Example. Determine the valence of N in HNO_3.

Solution. The total valence of oxygen is here $3x(-2) = -6$. The valence of hydrogen is + 1. To bring the total valence up to zero, the valence of the nitrogen must be $6 - 1 = 5$, $1 + 5 - 6 = 0$.

Chemical Equation for Reaction Involving Oxidation and Reduction

There are three general steps.

(a) We must know two things:

1. We must know what the products are.
2. We must know the valence of each element.

(b) We next calculate the oxidation and reduction in units of valence.

(c) Next state such numbers of formula weights (numbers of molecules) in both the materials and the products that the total oxidation will equal the total reduction.

Method illustrated. Lead dissolves in nitric acid to form lead nitrate, nitric oxide, and water. Complete the equation.

1st. With the products, thus: $Pb + HNO_3 = Pb(NO_3)_2 + NO + H_2O$. Compute oxidation and reduction. Here we notice that Pb changes from 0 valence to + 2, a gain of two.

2d. Nitrogen changes from + 5 (how obtained?) in nitric acid to + 2 (how obtained?) in nitric oxide, a loss of three.

3d. Balance oxidation and reduction. The least common multiple of 2 (the gain) and 3 (the loss in oxidation) is 6. For the lead to gain a total of 6 there must be considered 3 atomic weights; we write 3Pb. For the nitrogen in nitric oxide to lose a total of 6 there must be two molecular weights of nitric oxide considered; hence, we write 2NO. Our equation now stands, $3Pb + HNO_3 = Pb(NO_3)_2 + 2NO + H_2O$.

4th. Balance the metal. Three atomic weights of Pb will go to form three molecules of lead nitrate. Our equation now is, $3Pb + HNO_3 = 3Pb(NO_3)_2 + 2NO + H_2O$.

5th. Balance the acid radicals and water. The two molecular weights of NO require two of nitric acid, and

the six NO_3 radicals in $3Pb(NO_3)_2$ require six more of nitric acid; in all, eight of nitric acid. The eight atomic weights of hydrogen in $8HNO_3$ will form four molecular weights of water. Hence our equation completed is:

$$3Pb + 8HNO_3 = 3Pb(NO_3)_2 + 2NO + 4H_2O.$$

Verification. In order to verify the work, count the atomic weights of oxygen indicated in each member of the equation. If these are equal the work is probably correct.

Summary. In the solution of the example, then, we notice that there are five steps:

1. Write the products. (These must be known, or obtained from an authority.)

2. Compute the oxidation and reduction.

3. Balance the oxidation and reduction; i. e., state such numbers of formula weights that the total oxidation will equal the total reduction.

4. Balance the metal; i. e., 3 atomic weights of lead on one side of the equation necessarily require 3 on the other side.

5. Balance the acid radicals. Same as the argument for the metal.

Behavior of Lead with Analytical Reagents

Experiment 104.—*a.* Place a small piece of lead in the evaporating-dish. Add 10 or 15 c. c. of dilute C.P. nitric acid and warm gently. What gas is given off? Write the equation, as above, for the solution. Evaporate the solution of lead nitrate carefully to dryness, examine the crystals, and dissolve in about 10 c. c. of distilled water.

b. To the solution add a few drops of C.P. hydrochloric acid. Result?

From the "Method of Predicting Reactions" and

"Dissociations," write the equation for this reaction (see p. 152):

$$Pb(NO_3)_2 + HCl = ?$$

c. Filter and wash the precipitate with cold water, then place the precipitate in a half test-tube full of hot water. What result?

d. Divide the solution from *c* into four parts and test as follows:

1. To the first portion add a solution of hydrogen sulphide. Result?

Complete the equation: $PbCl_2 + H_2S = ?$

2. To the second portion add one drop of sulphuric acid. Result?

Complete the equation: $PbCl_2 + H_2SO_4 = ?$

3. To the third portion add a solution of potassium chromate, K_2CrO_4. Result?

Complete the equation: $PbCl_2 + K_2CrO_4 = ?$

4. To the fourth portion add a solution of potassium iodide. Result?

Complete the equation: $PbCl_2 + KI = ?$

When the last reaction is complete, heat the lead iodide solution to boiling. Result? Then hold the test-tube under flowing water from the hydrant. What result by cooling?

Summary. Lead nitrate dissolves in distilled water and is precipitated by hydrochloric acid. The lead chloride formed is slightly soluble in cold water, readily soluble in hot water. The solution in hot water forms a black precipitate with hydrogen sulphide, a white precipitate with sulphuric acid, a yellow precipitate of chrome yellow with potassium chromate, a deep yellow precipitate with

potassium iodide. Conformity to the above tests is conclusive proof for the presence of lead in a compound.

Each experiment should be summarized according to this plan.

SILVER

Occurrence. Silver occurs native and as the sulphide, Ag_2S. The latter is generally found with lead sulphide.

Metallurgy. The silver and lead of silver and lead sulphides are extracted by the same method as that employed in extracting lead alone from its ores. The product consists of silver dissolved in a large quantity of lead, and the problem for the metallurgist is how to separate the small amount of silver from the large amount of lead.

Pattinson's Method. By melting the mass of lead and silver and then allowing it to cool, crystals of almost pure lead separate from the fused mixture. These crystals are removed by a sieve-like ladle and the process is repeated. Finally, a product rich in silver is obtained. This is heated in contact with air, when the lead is oxidized and the silver remains unattached.

Zinc Method. This method consists in treating the molten mass of silver and lead with zinc. The zinc forms an alloy with the silver, and this alloy is heated with super-heated steam. This oxidizes the zinc, and the silver remains in the metallic state.

Solubility. Nitric acid is the best solvent for silver. Silver nitrate, nitric oxide and water are the products.

Equation:

$$xAg^{\circ} + xHNO_3 = xAgNO_3 + xNO + xH_2O.$$

Noting that silver is oxidized 1 in valence and that nitrogen is reduced 3 in valence (explain how), we see that, in order to have oxidation and reduction equal, 3 of silver nitrate and 1 of nitric oxide must be formed.

Balancing the metal, our equation will now stand:

$$3Ag + xHNO_3 = 3AgNO_3 + NO + xH_2O.$$

Next balancing the acid radicals, we see that the 3 radicals in $3AgNO_3$ and the one represented in NO will together require 4 of HNO_3. The 4 of H in HNO_3 will form $2H_2O$. Hence, our equation is:

$$3Ag + 4HNO_3 = 3AgNO_3 + NO + 2H_2O.$$

Verify as in the preceding experiment.

Behavior with Analytical Reagents

Experiment 105: Dissolve a small piece of silver in about 5 c. c. of dilute nitric acid. Evaporate carefully to dryness and examine the silver nitrate crystals. Silver nitrate is known also as "lunar caustic." Dissolve the silver nitrate in distilled water, add HCl, and notice the curdy white precipitate of silver chloride.

Equation: $AgNO_3 + HCl = ?$

Filter, wash with cold water, and then treat with much hot water. What result? How does this compare with the action of hot water on lead chloride?

How may lead chloride be separated from silver chloride?

Set aside part of the silver chloride where light will shine on it. Notice the change in color. Place the funnel with filter-paper and remaining silver chloride over a clean t. t. Add ammonium hydroxide. Result?

Equation:

$$3NH_4OH + 2AgCl = (NH_3)_2(AgCl)_2 + 3H_2O.$$

Add nitric acid. This neutralizes the ammonia. Result?

Equation:

$$(NH_3)_3(AgCl)_2 + 3NHO_3 = 3NH_4NO_3 + 2AgCl.$$

The solution of the chloride in ammonium hydroxide and the reprecipitation with nitric acid give conclusive proof for the presence of silver in a compound.

MERCURY

Mercury in Mercurous Compounds

Occurrence. Mercury occurs generally in the sulphide, cinnabar, HgS. It is obtained by roasting cinnabar. Vapors of mercury and sulphur dioxide are given off.

Solubility. For mercury, as well as for lead and silver, nitric acid is the best solvent. The products, if dilute acid and excess of mercury be used, are mercurous nitrate, nitric oxide, and water.

Observing the same principles as in the preceding experiment, complete the equation:

$$xHg + xHNO_3 = xHgNO_3 + xNO + xH_2O.$$

Behavior with Analytical Reagents

Experiment 106: Partly dissolve a small globule of mercury in about 5 c. c. of dilute nitric acid. Warm gently, but do not evaporate to dryness. Add water to the mercurous nitrate and divide in two portions. Set aside the first portion for the next experiment. To the second portion add hydrochloric acid. Result? $HgNO_3 + HCl = ?$ Filter, then treat with cold and hot water on the filter-paper. Result? Add NH_4OH. Compare with the action of ammonium hydroxide on silver chloride. How would you separate silver chloride from mercurous chloride?

Mercurous chloride is commonly known as calomel. The addition of ammonium hydroxide to calomel forms mercurous chloramide. NH_2Hg_2Cl. Compare mercurous chloramide with ammonium chloride, NH_4Cl.

Equation for the last reaction:

$2HgCl + 2NH_4OH = NH_2Hg_2Cl + NH_4Cl + 2H_2O.$

Precipitation of a solution with hydrochloric acid and blackening of the precipitate with ammonium hydroxide give conclusive proof for the presence of mercury in a compound.

Mercury in Mercuric Compounds

Take the portion of mercurous nitrate reserved in the preceding experiment and add to it 2 c. c. of nitric acid. Evaporate nearly to dryness. This insures complete oxidation of the mercury and also expels the excess of nitric acid.

Equation:

$xHgNO_3 + xHNO_3 = xHg(NO_3)_2 + xNO + xH_2O.$

Mercury is oxidized from a valence of 1 in $HgNO_3$ to a valence of 2 in $Hg(NO_3)_2$, a gain of 1. Nitrogen is reduced from a valence of 5 to 2, as in preceding cases. Balancing oxidation and reduction, we see that 3 mercuric nitrate would be formed in order to give a total oxidation of 3 in valence. Also that 1 of nitric oxide would be formed to give the reduction of 1 in valence. Balancing the metal and the acid radical we get:

$3HgNO_3 + 4HNO_3 = 3Hg(NO_3)_2 + NO + 2H_2O.$

Explain in detail.

Behavior with Analytical Reagents

Experiment 107: Dissolve the mercuric nitrate in distilled water. It may be necessary to add a drop of nitric acid to insure solution. To the solution of mercuric nitrate add a few drops of hydrochloric acid. If the mercury has been completely oxidized, no precipitate will form. Compare with action of hydrochloric acid on mercu*rous*

nitrate. Next pass hydrogen sulphide gas through the solution of mercuric nitrate. Result?

A progressive coloring through yellow and gray, followed by a dense black precipitate, is characteristic of the action of hydrogen sulphide on mercuric compounds. This is due to the formation first of basic salts of mercury. The final compound is mercuric sulphide, HgS. $Hg(NO_3)_2 + H_2S = ?$

Filter, wash, and test the solubility of a small portion of the mercuric sulphide in yellow ammonium sulphide, $(NH_4)_2S_2$. Next try the action of hot dilute nitric acid on the mercuric sulphide. Next pour over the mercuric sulphide some hot nitro-hydrochloric acid. The products are mercuric chloride, nitric oxide, free sulphur and water.

Equation:

$$xHgS + xHNO_3 + xHCl = xHgCl_2 + xS^\circ_2 + xNO + xH_2O.$$

1. The S is oxidized from -2 to 0, or for the molecule of S_2, a gain of 4. The nitrogen is reduced 3, as before. To make oxidation and reduction equal, then, 3 of $S_2(3 \times 4 = 12)$ and 4 of $NO(4 \times + 3 = + 12)$ will be formed. Our equation now stands:

$$xHgS + xHNO_3 + xHCl = xHgCl_2 + 3S_2 + 4NO + xH_2O.$$

2. Balance the S and the Hg. $3S_2 = 6$ of sulphur, which must be obtained from HgS. Hence, 6HgS. This makes 6 of mercury, which will give $6HgCl_2$ in the product. Our equation is now:

$$6HgS + xHNO_3 + xHCl = 6HgCl_2 + 3S_2 + 4NO + xH_2O.$$

3. Balance the chlorine, the nitric acid, and the water. $6HgCl_2$ contains 12 of chlorine, which means that 12HCl

are used. 4NO means that $4HNO_3$ are used. Total H = 16, making $8H_2O$. Hence,

$$6HgS + 4HNO_3 + 12HCl = 6HgCl_2 + 3S_2 + 4NO + 8H_2O.$$

Mercuric chloride, $HgCl_2$, is known as corrosive sublimate. Evaporate the solution nearly to dryness to expel the excess of acid, add water, and then add a solution of stannous chloride. This last reagent reduces the mercuric chloride to mercu*rous* chloride, insoluble, gray, proof for presence of mercury in a compound. Complete the equation:

$$xHgCl_2 + xSnCl_2 = xSnCl_4 + xHgCl, \textit{ gray precipitate.}$$

COPPER

Occurrence. Copper occurs native in the region of Lake Superior. It is found as the sulphide in Montana and Colorado, and as the carbonate in Arizona.

In other forms it occurs as copper pyrite, $CuFeS_2$; copper glance, Cu_2S; malachite, $Cu_2(OH)_2CO_3$.

Metallurgy. Copper oxide is reduced by heating with charcoal, or copper oxide and copper sulphide are mixed and heated. In the latter case free copper is obtained, while the sulphur and oxygen pass off as sulphur dioxide. The copper obtained by either method contains many impurities. This impure copper is at present refined almost entirely by the electrolytic method. For this purpose the metal is cast into plates 3 feet long by 18 inches wide by $\frac{3}{4}$ inch thick. These plates are made the positive poles, anodes, in an electrolytic cell. The cathodes are of pure electrolytic copper of the same length and breadth, and about $\frac{1}{30}$ inch in thickness. The anodes and cathodes are suspended opposite each other at a distance of 2 inches. All the anodes are connected, and similarly all the

cathodes. The electrolyte is a solution of copper sulphate acidulated with sulphuric acid. A current density of 10 to 20 amperes per square foot is used, and the fall of potential is from 0.2 to 0.5 of a volt in each cell. Copper obtained by this method shows a purity of 99.96 %.

Solubility. Cold dilute nitric acid readily dissolves copper. Hot concentrated sulphuric acid dissolves it equally well. The products are shown in the following equations, which should be completed by the student:

1. $xCu + xHNO_3 = xCu(NO_3)_2 + xNO + xH_2O$.
2. $xCu + xH_2SO_4 = xCuSO_4 + xSO_2 + xH_2O$.

Behavior with Analytical Reagents

Experiment 108: Place a small piece of copper in the evaporating-dish and add 10 c. c. nitric acid. When the copper has dissolved, evaporate to dryness but do not heat the residue. Dissolve the copper nitrate in water and add a few drops of hydrochloric acid.

Compare the reaction with the action of hydrochloric acid on the nitrates of lead, silver, and mercury.

Next pass hydrogen sulphide gas through the solution. Result? $Cu(NO)_2 + H_2S = ?$ Filter and wash. Try the action of yellow ammonium sulphide on a small portion of the precipitate. Next place the precipitate in a test-tube with about 10 c. c. hot dilute nitric acid. Compare the result with that of hot dilute nitric acid on mercuric sulphide. The products with the valence for each element oxidized or reduced are shown in the following equation. The student should complete the equation in accordance with principles outlined in the preceding experiments:

$xCuS + xHNO_3 = xCu(NO_3)_2 + xS^{\circ}_2 + xNO + xH_2O$.

(NOTE.—The gain in one molecule of S_2 is 4.)

Evaporate the solution of copper nitrate to expel excess of nitric acid, add a little water and then excess of ammonium hydroxide. Result?

Copper hydroxide, $Cu(OH)_2$, is first formed and then redissolved. The ammonia probably plays a part similar to that of water in process of crystallization, so that the formula for the copper compound may be written:

$$Cu(NO_3)_2,\ 4NH_3,\ H_2O.$$

Equation:

$$Cu(NO_3)_2 + 4NH_4OH = Cu(NO_3)_2,\ 4NH_3,\ H_2O, + 3H_2O.$$

The blue color with ammonium hydroxide is a good test for copper in all but mere traces. For detecting small quantities, proceed by the following method:

1. Acidulate the ammonium hydroxide solution with acetic acid. (With the blue solution, add acetic acid until the blue color disappears.)

$$Cu(NO_3)_2,\ 4NH_3,\ H_2O + 4H(C_2H_3O_2) = Cu(C_2H_3O_2)_2 + H_2O + 2NH_4(NO_3) + 2NH_4(C_2H_3O_2).$$

2. Add potassium ferrocyanide, $K_4Fe(CN)_6$. A reddish-brown precipitate is cupric ferrocyanide, $Cu_2Fe(CN)_6$, proof for presence of copper.

Complete: $(C_2H_3O_2) + K_4Fe(CN)_6 = ?$

IRON

Occurrence. The chief iron ores are hematite, Fe_2O_3; magnetite, Fe_3O_4; brown iron ore, $Fe_4O_3(OH)_6$; and spathic iron or siderite, $FeCO_3$. For a description of the metallurgy of iron a text-book should be consulted.

Solubility. Both hydrochloric and sulphuric acids dissolve iron, forming, if cold dilute acids be used, the ferr*ous* salt in each case and hydrogen. The student should record the equations.

With hot concentrated acids the ferric salts are formed; with sulphuric acid, a considerable reduction takes place, sulphur dioxide being formed.

Complete the following equations:

$xFe^{\circ} + xHCl$ (hot concentrated) $= FeCl_3 + H^{\circ}_2$.

$xFe^{\circ} + xH_2SO_4$ (hot concentrated) $= xFe_2(SO_4)_3 + xSO_2 + xH_2O$.

Cold dilute nitric acid dissolves iron to form ferrous nitrate. Hot, moderately dilute nitric acid forms ferric nitrate. Cold concentrated nitric acid does not dissolve iron.

Behavior with Analytical Reagents

Iron Forms Two Series of Compounds, Ferrous and Ferric

Experiment 109: Dissolve a little iron wire in dilute hydrochloric acid. Ferrous chloride and hydrogen are the products. Divide into two portions, and add ammonium hydroxide to the first. The precipitate is nearly white at first, ferrous hydroxide, but changes color, through green to reddish-brown rapidly as the iron passes to the ferric condition.

To the second portion add a few drops of concentrated nitric acid, and boil. Repeat the operation two or three times, then cool, and add ammonium hydroxide. Compare this result with that obtained by adding ammonium hydroxide to the original ferrous chloride. If complete oxidation has taken place, no trace of the green color will be seen.

Outline for Analysis of Iron Salts

Experiment 110: Ferrous sulphate ($FeSO_4 + 7H_2O$) is commonly known as "green vitriol," or "copperas." (What is "white vitriol"? "blue vitriol"? "oil of vitriol"?)

a. Dissolve 2 or 3 grams of ferrous sulphate in about 20 c. c. of water, pour about 5 c. c. of the solution into a test-tube, and add a few drops of hydrochloric acid. Result? Compare with the action of hydrochloric acid on first-group metals.

b. Pass hydrogen sulphide through the solution containing the ferrous sulphate and hydrochloric acid. Result? Compare with the action of hydrogen sulphide on the second-group salts already studied; i. e., mercuric and copper salts. Does iron evidently belong to the second group?

c. Boil strongly to expel the hydrogen sulphide, then add a few drops of nitric acid and boil again to oxidize the iron.

Equation:

$$xFeSO_4 + xHNO_3 = xFe_2(SO_4)_3 + xFe(NO_3)_3 + xNO + xH_2O.$$

In accordance with the principles of oxidation and reduction in preceding experiments, complete the above equation. Note that iron is oxidized from 2 to 3, and that the nitrogen of nitric oxide is reduced from 5 to 2.

d. To the ferric sulphate solution add ammonium chloride, and immediately excess of ammonium hydroxide. Result? Complete the equation:

$$xFe_2(SO_4)_3 + xNH_4OH = (NH_4)_2SO_4 + xFe(OH)_3.$$

e. Filter, wash, and add cold potassium hydroxide to a portion of the precipitate. Result? Next place the funnel over a clean test-tube and add cold dilute hydrochloric acid. Result? Complete the equation:

$$Fe(OH)_3 + HCl = ?$$

f. To the solution obtained in *e* add one drop of solution of potassium sulphocyanide, KCNS. Note result, and

then add more KCNS. With ferric salts, KCNS forms a cherry-red solution of ferric sulphocyanide, $Fe(CNS)_3$.

Equation: $FeCl_3 + K(CNS) = ?$

To Distinguish Between Ferrous and Ferric Compounds

Experiment 111: Divide a solution of ferrous sulphate into two portions.

a. To the first portion add a solution of potassium ferricyanide, $K_3Fe(CN)_6$. The dark-blue precipitate is ferrous ferricyanide, $Fe_3(Fe(CN)_6)_2$.

Equation: $FeSO_4 + K_3Fe(CN)_6 = ?$

b. Oxidize the second portion of ferrous sulphate to ferric sulphate by boiling with nitric acid. See *c* above. Then cool, and dilute somewhat, and add potassium ferricyanide as in *a*. No precipitate is formed, but the solution is colored brown or green by the formation of ferric ferricyanide, $Fe(Fe(CN)_6)$.

$$Fe_2(SO_4)_3 + 2K_3Fe(CN)_6 = 3K_2SO_4 + 2FeFe(CN)_6.$$

Prussian Blue

Experiment 112: To a solution of ferric chloride or ferric sulphate add potassium ferrocyanide, $K_4Fe(CN)_6$. (See experiment with copper.) The precipitate is ferric ferrocyanide, Prussian blue, $Fe_4(Fe(CN)_6)_3$.

$$4FeCl_3 + 3K_4Fe(CN)_6 = 12KCl + Fe_4(Fe(CN)_6)_3.$$

ALUMINIUM

Occurrence. Aluminium is contained in the important minerals, feldspar, granite, cryolite, clay, mica.

The composition of feldspar is represented by the formula, $KAlSi_3O_8$; it is the potassium aluminium salt of trisilicic acid. Granite is composed of quartz, feldspar, and mica in varying proportions. A decomposition

product of granite is clay, the formula being $Al_4(SiO_4)_3 + 4H_2O$. The name mica applies to a large number of minerals composed of aluminium, silicon, oxygen, and magnesium or potassium. The simplest form of mica is $KAlSiO_4$, a salt of orthosilicic acid. Cryolite is a salt of sodium aluminium and fluorine, Na_3AlF_6. Corundum is Al_2O_3; when found in red transparent crystals it is called ruby, and the opaque variety is emery. Banxite is Al_2O_3, $2H_2O$.

Metallurgy. Aluminium differs from all other metals used in the metallic state, in that at present it is produced by the electrolytic method only. A bath of cryolite is maintained in the molten condition by the passage of an electric current. The cryolite is contained in an iron vessel which is itself the negative electrode; carbon rods suspended in the cryolite form the positive electrodes. Pulverized corundum, Al_2O_3, is added to the molten cryolite; it dissolves immediately and becomes the electrolyte.

Metallic aluminium separates in the molten condition at the bottom of the iron vessel, while oxygen is liberated at the carbon electrodes which gradually waste away.

The most extensive plant for the manufacture of aluminium is at the Niagara Falls Works of the Pittsburgh Reduction Company.

Solubility. Hydrochloric acid is the best solvent for aluminium. Aluminium chloride ($AlCl_3$) and hydrogen are the products. Complete:

$$Al + HCl = AlCl_3 + H_2.$$

Sulphuric and nitric acids, hot, slowly dissolve aluminium, forming the corresponding salts. Complete:

$$Al + H_2SO_4 = Al_2(SO_4)_3 + SO_2 + H_2O.$$

Hot solutions of potassium or sodium hydroxide dis-

solve aluminium, forming the so-called aluminates, $KAlO_2$, and hydrogen. Complete:

$$Al^\circ + KOH + H_2O = KAlO_2 + H^\circ_2.$$

Behavior with Analytical Reagents

Experiment 113: Dissolve a small piece of aluminium in hydrochloric acid.

Equation: $xAl + xHCl = AlCl_3 + xH_2$.

Evaporate nearly to dryness, cool, and dissolve in distilled water. Add a few drops of hydrochloric acid. Result? Inference? Next pass hydrogen sulphide gas through the same solution. Result? Inference? Boil to expel hydrogen sulphide, add a few drops of nitric acid, a little ammonium chloride, and immediately, excess of ammonium hydroxide. What result is obtained with this last reagent? Complete:

$$AlCl_3 + NH_4OH = Al(OH)_3 + NH_4Cl.$$

Filter, wash with cold water, and transfer some of the precipitate to a test-tube. Add excess of potassium hydroxide, *agitate.* Compare with action of KOH on ferric hydroxide. Inference? Complete:

$$Al(OH)_3 + KOH = KAlO_2 + H_2O.$$

Next heat gradually to boiling. Result? Add hydrochloric acid to a slightly acid reaction. Complete the equation:

$$xKAlO_2 + xHCl = xH_2O + xKCl + xAlCl_3.$$

Finally, add excess of ammonium carbonate to the last solution. Result?

Equation:

$$xAlCl_3 + x(NH_4)_2CO_3 + xH_2O = xAl(OH)_3 + xNH_4Cl + xCO_2.$$

A gelatinous precipitate at this point is $Al(OH)_3$, conclusive proof for the presence of aluminium.

Compounds of Aluminium

Alum. The alums are double salts of the sulphate of some alkali metal or ammonium, K_2SO_4, Na_2SO_4, $(NH_4)_2SO_4$, combined with the sulphate of aluminium, ferric iron, or chromium, $Al_2(SO_4)_3$, $Fe_2(SO_4)_3$, $Cr_2(SO_4)_3$.

Common alum is ordinarily written, $KAl(SO_4)_2$; iron alum is $KFe(SO_4)_2$. Sodium or ammonium may be substituted for the potassium in any of these formulas.

Ultramarine. This coloring matter was originally obtained by powdering the mineral lapis lazuli. It consists of a silicate of aluminium and sodium together with a polysulphide of sodium. Ultramarine is now prepared artificially on a large scale by melting together kaolin, anhydrous sodium carbonate, and sulphur.

Spinel. Aluminium forms another hydroxide, $HAlO_2$. In many respects this resembles an acid. If the hydrogen of $HAlO_2$ be replaced by magnesium, we have the most common of the spinel group, magnesium aluminate, $Mg(AlO_2)_2$. The magnesium may be replaced by divalent iron, and the aluminium by chromium. In that case we have $Fe(CrO_2)_2$, known as chromic iron.

CHROMIUM

Occurrence. This element is comparatively rare, and occurs almost always in the mineral, chromic iron, $Fe(CrO_2)_2$. See preceding experiment. It is sometimes found as the oxide.

Metallurgy. Chromium is prepared by heating a mixture of chromium oxide, Cr_2O_3, and powdered carbon in the electric furnace. The temperature of the ordinary furnace in which carbon is the fuel does not exceed 2,000° C., and is not sufficiently high to reduce chromic oxide. In the electric furnace, however, a temperature

of 3,500° C. is easily obtained. By this means chromium containing from 1 to 10 % of carbon is obtained. This product is fused with lime, and pure chromium is obtained, the carbon uniting itself with the calcium to form carbide.

Another method of preparing chromium is by fusing a mixture of chromic oxide and powdered aluminium. The mass is ignited by a fuse composed of a mixture of pulverized aluminium and barium dioxide, in which a strip of magnesium has been placed so that the mixture may be kindled.

Chromium is a gray metal of a brilliant luster, and, though hard, may be filed and polished without difficulty. Alloyed with iron, it is of importance in making projectiles, as the alloy is both hard and tough.

Compounds of Chromium

In chromic acid, H_2CrO_4, and chromates, as K_2CrO_4, chromium shows non-metallic properties and closely resembles sulphur. Chromic anhydride (CrO_3) is a bright scarlet solid. Compare with sulphuric anhydride.

Potassium chromate (K_2CrO_4) is a bright yellow, easily soluble compound. If a solution of it is warmed slightly and a small amount of nitric acid carefully added, the color gradually changes to red by the formation of potassium dichromate, $K_2Cr_2O_7$. Upon evaporation this compound is obtained in the crystalline form.

Potassium dichromate may be considered as formed by the addition of chromic anhydride to potassium normal chromate, $K_2CrO_4 + CrO_3 = K_2Cr_2O_7$.

Chromium forms another series of compounds in which the element plays the part of a metal. Among these may be mentioned chrome alum, $KCr(SO_4)_2$, chromic sulphate, $Cr_2(SO_4)_2$, and chromium hydroxide.

Behavior with Analytical Reagents

Experiment 114: Dissolve some chrome alum in water and add a little hydrochloric acid. Result? To the solution next add hydrogen sulphide, either as the gas or as the solution. Result? Inference? Next remove the hydrogen sulphide by boiling, add a little nitric acid, then ammonium chloride, and immediately excess of ammonium hydroxide. Result? Complete the equation:

$$xCr_2(SO_4)_3 + xNH_4OH = xCr(OH)_3 + x(NH_4)_2SO_4.$$

Filter, and wash with a little cold water. Place some of the chromium hydroxide in a test-tube and add an excess of potassium hydroxide. Result? Compare with action of potassium hydroxide on ferric hydroxide. Complete:

$$Cr(OH)_3 + KOH = KCrO_2 + H_2O.$$

The solution contains potassium chromate. Boil vigorously for two or three minutes. Result? Hot water causes the addition of hydroxyl groups to the chromium, so that chromium hydroxide is again formed. Compare with the action of hot water on potassium aluminate. Question: How may chromium hydroxide be separated from a mixture of potassium chromite with potassium aluminate? Complete the equation:

$$xKCrO_2 + xH_2O\,(\text{boiling}) = xCr(OH)_3 + xKOH.$$

Filter, and transfer part of the precipitate to a small piece of platinum foil, or a piece of sheet iron may be used for a rough test. Add about $\frac{1}{2}$ gram of a mixture of equal parts of potassium nitrate and sodium carbonate. Such a mixture is called a "fusing mixture," and is used for oxidation when fusing is necessary. Heat the chromium oxide with the fusing mixture on the platinum foil in the oxidizing flame before the blow-pipe until a yellow color appears.

This is evidence of the formation of potassium and sodium chromates in the mass. Complete the following:

$$xCr(OH)_3 + xKNO_3 + xNa_2CO_3 = xK_2CrO_4 + xNa_2CrO_4 + xNO + xH_2O + xCO_2.$$

Dissolve the mass in distilled water and add acetic acid until the effervescence (what causes it?) ceases. Divide into two portions. To the first portion add a little lead nitrate. Result?

$$K_2CrO_4 + Pb(NO_3)_2 = ?$$

To the second portion add silver nitrate. A red precipitate in the last case is silver chromate, red, conclusive proof for the presence of chromium.

$$K_2CrO_4 + 2AgNO_3 = ?$$

Iron, aluminium, and chromium belong to the third group in analysis. At this point the student should prepare a summary in tabular form of the experiments with iron, chromium, and aluminium. Ammonium hydroxide is called the third-group reagent.

MANGANESE

Occurrence. Manganese accompanies nearly all iron ores. Its chief ore is pyrolusite, MnO_2. It occurs also as manganese spar, $MnCo_3$; manganese blende, MnS.

Metallurgy. Metallic manganese is prepared by electrolysis of manganese chloride; another method is by ignition of manganese oxide with metallic aluminium. (Compare preparation of chromium by similar means.)

Solubility. Manganese dissolves in dilute acids to form manganous salts. Manganese dioxide (MnO_2) dissolves in hydrochloric acid to form manganous chloride, $MnCl_2$, free chlorine, Cl_2, and water. Complete the equation:

$$xMnO_2 + xHCl = MnCl_2 + Cl_2 + H_2O.$$

If sulphuric acid and a chloride are used the action is similar.

$$xNaCl + xH_2SO_4 + xMnO_2 = MnSO_4 + Na_2SO_4 + Cl_2 + H_2O.$$

Behavior with Analytical Reagents

Experiment 115: Place 1 g. manganese dioxide in the evaporating-dish, add 5 c. c. of concentrated sulphuric acid, place under the hood and warm gently.

$$xMnO_2 + xH_2SO_4 = xMnSO_4 + xH_2O + xO_2.$$

Evaporate nearly to dryness, cool, and dissolve in distilled water. Add a few drops of hydrochloric acid. Result?

$$MnSo_4 + HCl = ?$$

Pass hydrogen sulphide gas through this acid solution of $MnSO_4$. Result?

$$MnSO_4 + H_2S = ?$$

(NOTE.—This is an *acid* solution.) Boil out the H_2S thoroughly, add a few drops of nitric acid. Result?

$$MnSO_4 + HNO_3 = ?$$

Next add excess of ammonium chloride. A double salt of ammonium and manganese is formed, although no visible action takes place.

$$MnSO_4 + 2NH_4Cl = MnSO_4, 2NH_4Cl.$$

Next add a strong excess of ammonium hydroxide. Result?

$$MnSO_4, 2NH_4Cl + NH_4OH = ?$$

What separation does this suggest? Note that this solution is alkaline. Next pass hydrogen sulphide gas through the solution in the alkaline condition. Result?

$$MnSO_4, 2NH_4Cl + xNH_4OH + xH_2S = MnS + x(NH_4)_2SO_4 + 2NH_4Cl + H_2O.$$

Filter, and wash thoroughly and pour cold dilute hydrochloric acid over the ppt. Result?

$$MnS + HCl = ?$$

Boil the solution of $MnCl_2$ *thoroughly* to expel *all* the H_2S, then add excess of potassium hydroxide. Result?

$$MnCl_2 + xKOH = xKCl + Mn(OH)_2.$$

Place a small portion of the precipitate on some iron or platinum foil, add ½ g. of fusing mixture ($KNO_3 + Na_2CO_3$; see experiment with chromium), and fuse strongly in the oxidizing flame before the blow-pipe. The manganous hydroxide is oxidized to the green mass of potassium manganate. Final test. Complete the equation:

$$Mn(OH)_2 + xKNO_3 + xNa_2CO_3 = K_2MnO_4 + Na_2MnO_4 + CO_2 + H_2O.$$

Dissolve the K_2MnO_4 in as little water as possible, and add two drops HNO_3.

$$xK_2MnO_4 + xHNO_3 = KMnO_4 + KNO_3 + NO + H_2O.$$

ZINC

Occurrence. Zinc is found chiefly as Smithsonite, $ZnCO_3$, and as zinc blende, ZnS.

Metallurgy. The preparation of zinc consists of two principal steps. The carbonate or sulphide is first roasted, forming zinc oxide (ZnO) in either case. The zinc oxide is mixed with powdered charcoal and heated to a white heat. The oxide is reduced and the zinc vaporized. The metallic zinc vapor is condensed in suitable receivers.

Solubility. Pure zinc dissolves very slowly in acids or alkalis, unless in contact with Cu, Pb, or a less positive metal. The impurities in commercial zinc enable it to dissolve readily in acids and alkalis.

Behavior with Analytical Reagents

Experiment 116: Dissolve a small piece of zinc in dilute nitric acid.

$$xZn + xHNO_3 = Zn(NO_3)_2 + NO + H_2O.$$

Evaporate nearly to dryness, dissolve in distilled water, and add a little hydrochloric acid. Result?

$$Zn(NO_3)_2 + HCl = ?$$

Next pass hydrogen sulphide gas through the solution. Result? Inference?

$$Zn(NO_3)_2 + H_2S \text{ (acid solution)} = ?$$

Boil out the H_2S thoroughly, add a few drops of HNO_3, then NH_4Cl. Result?

$$Zn(NO_3)_2 + NH_4Cl = ?$$

Add ammonium hydroxide slowly. Result?

$$Zn(NO_3)_2 + xNH_4OH = Zn(OH)_2 + NH_4NO_3.$$

Next add *excess* of NH_4OH. Result?

$$Zn(OH)_2 + 2NH_4OH = (NH_4)_2ZnO_2 + 2H_2O.$$

A limited amount of ammonium hydroxide precipitates $Zn(OH)_2$, but an excess of ammonium hydroxide dissolves the $Zn(OH)_2$ first precipitated and forms ammonium zincate. What property of zinc does this illustrate? What separation is suggested?

Next pass H_2S gas through the alkaline solution. Result?

$$(NH_4)_2ZnO_2 + H_2S = ZnS + 2NH_4OH.$$

Filter, wash, and add cold dilute HCl. Result?

$$ZnS + HCl = ?$$

Boil *very thoroughly* for several minutes to expel *all* the H_2S. Add a small portion of potassium hydroxide. Result?

$$ZnCl_2 + xKOH = Zn(OH)_2 + xKCl.$$

Now add excess of KOH. Result?

$$Zn(OH)_2 + 2KOH = K_2ZnO_2 + 2H_2O.$$

Excess of KOH redissolves zinc hydroxide, forming potassium zincate.

To the solution add hydrogen sulphide; a white or gray precipitate is zinc sulphide, proof for presence of zinc.

$$K_2ZnO_2 + H_2S = ZnS + 2KOH.$$

MAGNESIUM

Occurrence. Magnesium is found chiefly as magnesite, $MgC0_3$; as dolomite, $MgCa(CO_3)_2$; as epsom salts, $MgSO_4$, $7H_2O$; as the double chloride, KCl, $MgCl_2$, $6H_2O$, called carnollite.

Metallurgy. Magnesium is obtained principally by the electrolysis of the fused salt, KCl, $MgCl_2$, $6H_2O$. Magnesium chloride ($MgCl_2$) in water solution is largely decomposed upon evaporation, magnesium oxide (MgO) and gaseous hydrochloric acid resulting. But $KClMgCl_2$, $6H_2O$ can be dehydrated without decomposition. In the preparation of magnesium, the anhydrous KCl is fused in a steel vessel, which forms the cathode.

The anode is a carbon rod suspended in a porcelain cylinder open at the bottom. Chlorine separates at the anode and is conveyed away by a pipe from the porcelain cylinder. Magnesium separates at the cathode and floats on the surface of the fused mass. It is protected from contact with the atmosphere by an air-tight cover to the steel vessel.

Solubility. Magnesium is soluble in acids, including CO_2 in water, with evolution of hydrogen.

$$Mg + CO_2 + H_2O = MgCO_3 + H_2.$$

Behavior with Analytical Reagents

Experiment 117: Dissolve a piece of magnesium in dilute sulphuric acid.

$$Mg + H_2SO_4 = ?$$

Evaporate to dryness and dissolve in distilled water. Add HCl. Result?

$$MgSO_4 + HCl = ?$$

Pass H_2S through the solution. Result? Inference?

$$MgSO_4 + H_2S = ?$$

Boil out the H_2S, add HNO_3. Result?

Add excess of NH_4Cl. No visible result occurs, but a double chloride is formed.

$$MgSO_4 + NH_4Cl = MgSO_4,\ NH_4Cl.$$

Add excess of NH_4OH. Result? Add H_2S. Result? Inference? Boil out the H_2S and add NH_4OH and $(NH_4)_2CO_3$. Result? Inference?

To the alkaline solution add disodium hydrogen phosphate. A pearly gray precipitate is proof for the presence of magnesium ammonium phosphate.

$$MgSO_4,\ NH_4Cl + xNH_4OH + Na_2HPO_4 = MgNH_4PO_4 + Na_2SO_4 + NH_4Cl + H_2O.$$

COBALT

Cobalt is similar to iron in appearance, harder than Fe or Ni. It is malleable, very ductile, and the most tenacious of any metal, the wire being about twice as strong as iron wire. It is attracted by the magnet and can be made magnetic, retaining (unlike steel) its magnetism at a white heat.

Solubility. HCl and H_2SO_4 dissolve Co slowly. HNO_3 is the best solvent, forming $Co(NO_3)_2$. Reactions.

Behavior with Analytical Reagents

Experiment 118:

1. $Co(NO_3)_2 + HCl = ?$
2. $Co(NO_3)_2 + H_2S = ?$
3. Boil, add HNO_3, NH_4Cl, and NH_4OH. No ppt.
4. Add H_2S. $Co(NO_3)_2 + H_2S$ (alkaline solution $= ?$)
5. Filter, add cold dilute HCl. MnS and ZnS dissolve at this point. CoS does not.
6. Form a very small loop at the end of the platinum wire, heat to redness, dip into powdered borax and heat again. A clear "borax bead" is formed. While the bead is hot, touch it to the ppt. of CoS in (5), and heat again. A brilliant blue color is imparted to the bead by the Co.

Microcosmic salt may be used in place of borax. Borax is better.

If nickel be present, see test for nickel for the separation of Co.

NICKEL

Behavior with Analytical Reagents

Experiment 119: Proceed as in the analysis of cobalt until the black precipitate is obtained in Group 3 B. Dissolve in nitro-hydrochloric acid. Evaporate, and add an excess of nitroso—B—naphthol in acetic solution. This precipitates cobalt, while nickel is left in solution. Filter, and add ammonium hydroxide to the filtrate until alkaline. Filter again, and add hydrogen sulphide to the filtrate. A black precipitate (NiS) indicates nickel.

BARIUM

Barium is a white metal, stable in dry air, but rapidly oxidizing in moist air.

Preparation. By electrolysis of the fused chloride. By electrolysis of the sulphate or carbonate mixed with Hg and HgO, and then distillation of the amalgam.

Behavior with Analytical Reagents

Experiment 120:

a. To a solution of $Ba(NO_3)_2$ add HCl. $Ba(NO_3)_2 + HCl = ?$

b. Pass H_2S gas through the solution. Result? $Ba(NO_3)_2 + H_2S = ?$

c. Boil, add HNO_3, NH_4Cl, and NH_4OH. Result? Conclusion?

d. Pass H_2S gas through this ammoniacal solution. Result? Conclusion?

e. Boil, add NH_4OH and then $(NH_4)_2CO_3$. Result?

$$Ba(NO_3)_2 + (NH_4)_2CO_3 = ?$$

f. Filter, add $HC_2H_3O_2$. $BaCO_3 + HC_2H_3O_2 = ?$

g. Add $K_2Cr_2O_7$. Result?

$$Ba(C_2H_3O_2)_2 + K_2Cr_2O_7 + H_2O = BaCrO_4 + 2KC_2H_3O_2 + H_2CrO_4.$$

h. Filter, and dissolve $BaCrO_4$ in HCl. $BaCrO_4 + HCl = ?$

i. Add one drop of H_2SO_4 to solution formed in preceding step. A white ppt., $BaSO_4$, insoluble in HNO_3 or HCl, is proof for presence of barium.

$$BaCl_2 + H_2SO_4 = ?$$

STRONTIUM

Separation and Tests for Strontium

Experiment 121: To the filtrate from the zinc group add ammonium hydroxide and ammonium carbonate. Warm, but do not boil. Precipitate is $SrCO_3$. Filter,

wash, and add dilute acetic acid on the filter-paper. Solution is $Sr(C_2H_3O_2)_2$. To a small portion of the solution add potassium bichromate, and if a precipitate appears add the bichromate to the whole solution. This precipitate is due to the presence of barium. Filter, and to the filtrate add ammonium carbonate. The ppt. is $SrCO_3$, and must be filtered to separate it from the colored solution. Dissolve in acetic acid, and to the solution add a solution of $CaSO_4$. Boil, and let stand ten minutes. A ppt. indicates $SrSO_4$.

Test another portion of the solution on a platinum wire by the flame.

CALCIUM

Separation and Test for Calcium

Experiment 122: Proceed exactly as in the analysis for strontium until the barium is separated by adding potassium dichromate to the acetic acid solution. Then add ammonium carbonate; ppt. is $SrCO_3$ or $CaCO_3$. Filter and wash, and dissolve in acetic acid. To the solution add potassium sulphate, boil, and allow to stand ten minutes to insure the precipitation of all strontium. Filter, and to the filtrate (or unprecipitated solution) add ammonium oxalate. A white precipitate, insoluble in acetic, soluble in hydrochloric acid, indicates calcium, as CaC_2O_4.

ARSENIC

From the Analytical Standpoint

Experiment 123: Dissolve a small portion of As_2O_3 in HCl, dilute, and pass H_2S gas through the solution. Result? To what group does this belong?

a. $As_2O_3 + xHCl + 3H_2S = As_2S_3 + xHCl + 3H_2O$.

b. Filter, add ammonium sulphide. Result?

$$As_2S_3 + 2(NH_4)_2S = (NH_4)_4As_2S_5.$$

Compare this action with the action of $(NH_4)_2S$ on CuS and HgS. What separation does this suggest?

c. Add dilute HCl to the last solution. Result?

$$(NH_4)_4As_2S_5 + 4HCl = As_2S_3 + 4NH_4Cl + 2H_2S.$$

d. Filter, and add $(NH_4)_2CO_3$ to the ppt. of As_2O_3. Result?

The solution is a mixture of $(NH_4)_3AsO_3$ and $(NH_4)_3AsS_3$.

Finally, to the last solution add HCl. A yellow color is proof for the presence of arsenic.

ANTIMONY

From the Analytical Standpoint

Experiment 124:

a. Dissolve some Sb_2O_3 in dilute HCl.

$$Sb_2O_3 + 2HCl = 2SbOCl + H_2O.$$

This compound is called antimonyl chloride. Pass H_2S gas through the solution. Result? What group?

b. $SbOCl + xH_2S = Sb_2S_3 + xHCl$.

c. Filter, and add $(NH_4)_2S_2$, yellow ammonium sulphide. Result?

$2Sb_2S_3 + 6(NH_4)_2S = S_2 + 4(NH_4)_3SbS_4$, ammonium sulpho antimon*ate*. Compare with action of ammonium sulphide on CuS, HgS.

d. Add dilute HCl to the solution. Result? A reddish-orange ppt. at this point is proof for presence of antimony. This precipitate is Sb_2S_5, and is almost insoluble in $(NH_4)_2Co_3$, distinction from As_2S_3, and this furnishes a convenient means for a partial separation of As_2S_3 from Sb_2S_5.

BISMUTH

Valence two, three, and five, usually three.

Occurrence. It is comparatively rare, usually occurs native, or as the oxide or sulphide. Generally found in connection with cobalt ores.

Preparation. The rock containing bismuth and cobalt is roasted to remove sulphur and arsenic (generally present). Then the mass is fused with charcoal, and the bismuth settles to the bottom, cobalt above. The separation is effected mechanically.

Solubility. Nitric acid is the best solvent.

$Bi + HNO_3 = Bi(NO_3)_3 + NO + H_2O$. (Balance.)

Behavior with Analytical Reagents

Experiment 125:

a. $Bi(NO_3)_3 + H_2O = 2HNO_3 + BiONO_3$. Basic nitrate precipitate, but soluble upon boiling and adding a few drops of HNO_3.

b. $BiONO_3 + HCl$ gives a ppt., BiOCl, like Pb, Ag, and Hg, but this ppt. dissolves upon adding HNO_3, distinction from Pb, Ag, and Hg.

c. $BiOCl + H_2S = ?$ i. e., color, etc. $Bi_2S_3 + H_2O + HCl$. (Balance.)

d. Filter, add $(NH_4)_2S_2$ to *part* of *ppt.* Result? Inference?

Add hot dilute HNO_3, $Bi_2S_3 + HNO_3 = Bi(NO_3)_3 + NO + S_2 + H_2O$.

e. Concentrate to expel excess of HNO_3, cool, add $NH_4OH = ?$

$Bi(NO_3)_3 + NH_4OH = Bi(OH)_3 + NH_4NO_3$. Filter.

f. For final test for Bi: At this point prepare some

hot potassium stannite as follows: To solution of $SnCl_2$ add KOH until ppt. first formed *just barely dissolves:*

$$SnCl_2 + 4KOH = K_2SnO_2 + 2KCl + 2H_2O.$$

g. To the ppt. of $Bi(OH)_3$, see *e,* add hot K_2SnO_2, or better, put Bi(OH) in test-tube and pour K_2SnO_2 over it. Result? Final test:

$Bi(OH)_3 + K_2SnO_2 = Bi^0 + K_2SnO_3 + H_2O$. (Balance.)

CADMIUM

Occurrence. The sulphide (CdS, greenockite) occurs in Greenland, Scotland, and Pennsylvania; also to considerable extent with zinc ores.

Preparation. Reduced by carbon and separated from zinc by distillation, the cadmium being more volatile.

Solubility. Soluble in hydrochloric, sulphuric, or nitric acids, best in nitric acid.

Reactions with Analytical Reagents

Experiment 126:

a. $Cd(NO_3)_2 + HCl = ?$

b. $Cd(NO_3)_2 + H_2S = ?$

c. To a part of ppt. in *b* add $(NH_4)_2S_2 = ?$

d. To ppt. in *b* add hot dilute $HNO_3 = ?$

$$CdS + HNO_3 = Cd(NO_3)_2 + S_2{}^0 + NO + H_2O.$$

(Balance.)

Compare with Cu and Bi.

e. To solution from *d* add $NH_4OH = ?$

Compare with Cu and Bi.

f. To alkaline solution from *e* add $H_2S = ?$

NOTE.—If we add NH_4OH to a mixture of $Bi(NO_3)_3$, $Cu(NO_3)_2$, and $Cd(NO_3)_2$, the Bi is precipitated as $Bi(OH)_3$, white, while Cu and Cd are held in solution, the

Cu imparting a blue color. If we add KCN to the solution, until the blue color disappears, the Cd may then be precipitated as yellow CdS by adding H_2S. If no Cu be present, Cd may be at once precipitated as CdS by adding H_2S to the ammonium solution.

TIN

Tin dissolves in hot HCl, dilute HNO_3 forming stannous salts, Sn^{II}. Nitro-hydrochloric acid forms the stannic salt, Sn^{IV}.

Stannous salts with NaOH or KOH form stannous hydroxide, $Sn(OH)_2$, which is capable of acting either as a base or a weak acid. With excess NaOH it forms Na_2SnO_2; it is a strong reducing agent. Mercuric (Hg^{II}), silver (Ag^{I}), or ferric (Fe^{III}) compounds readily produce this change. Hence, a good test for tin is based on this reaction.

Behavior with Analytical Reagents

Experiment 127: Add KOH to the solution of $SnCl_2$ until the precipitate first formed redissolves.

$$SnCl_2 + 4KOH = K_2SnO_2 + 2KCl + 2H_2O.$$

Add a cold ammoniacal solution of $AgNO_3$, (1 volume $\frac{n}{4}AgNO_3$ to 15 volumes NH_4OH). A black precipitate of metallic silver will be formed, the tin being oxidized; proof for tin.

HIGHLY OXIDIZED CHEMICALS

Individual Tests

Experiment 128: *Potassium chlorate.* Place three or four crystals in a dry test-tube. A few drops of concentrated sulphuric acid poured on the crystals will produce decrepitation. Use only very few crystals.

Experiment 129: *Potassium dichromate, red.* Place a piece the size of a pea in an evaporating-dish and warm with hydrochloric acid. Add 2 c. c. of alcohol, and warm carefully. The red color will disappear, and chromium chloride, green, be formed. The chromium may be tested by the usual method.

Experiment 130: *Potassium normal chromate, yellow.* Proceed exactly as with red chromate. The above compounds may have their potassium replaced by sodium, ammonium, or other metals. Then the usual test for the metallic part is always applied.

Experiment 131: *Mercuric oxide, red.* Dissolve in hot, dilute nitric acid, and test by the usual method.

Experiment 132: *Red oxide of lead,* Pb_3O_4. Place a piece the size of a pea in an evaporating-dish, add nitric acid, and heat to boiling. Some PbO_2, black, will be formed. Add 2 c. c. of alcohol and heat carefully. The black color will disappear, a colorless solution of lead nitrate being formed. Test by the regular method.

Experiment 133: *Litharge,* PbO, *light, yellowish-brown.* Dissolve in hot nitric acid and test as above.

Experiment 134: *Potassium permanganate,* $KMnO_4$, *dark purple crystals.* Boil with hydrochloric acid *carefully;* slight explosions may occur, hence we use only a few crystals. Manganese chloride and free chlorine are formed.

Experiment 135: *Manganese dioxide, black.* Dissolve a small amount in hydrochloric acid. Manganese chloride and free chlorine are formed.

Experiment 136: *Copper oxide, black.* Dissolve in nitric acid.

Experiment 137: *Metallic iron.* Particles of metallic iron may be removed from a mixture by means of a magnet. Dissolve, and test by the usual method for iron.

TESTS FOR ACID IONS

Hydrochloric Acid and Chlorides

Experiment 138: Interfering metals are removed by treating the solution with sodium carbonate and filtering. To the filtrate add silver nitrate. The precipitate is silver chloride: filter, place the funnel over a clean test-tube and add ammonium hydroxide. The precipitate dissolves, forming ammonia silver chloride, and is reprecipitated by nitric acid, forming silver chloride again. This is proof for the presence of a chloride.

Carbonates

Experiment 139: Place about 1 g. of the original material in test-tube provided with a stopper and 2-bend delivery-tube. Add $HC_2H_3O_2$ to the substance, and pass the gas evolved through lime-water. A precipitate is $CaCO_3$; proof that CO_2 is passing into the lime-water. How does this prove the presence of a carbonate?

Equations:

$Na_2CO_3 + 2HC_2H_3O_2 = 2NaC_2H_3O_2 + H_2O + CO_2$.

$Ca(OH)_2 + CO_2 = CaCO_3$. If the carbon dioxide is passed through the lime-water after the calcium is all precipitated, the $CaCO_3$ begins to redissolve, forming $Ca(HCO_3)_2$. $CaCO_3 + H_2O + CO_2 = Ca(HCO_3)_2$. Upon boiling, this precipitate again $Ca(HCO_3)_2 + \text{boiling} = CaCO_3 + H_2O + CO_2$.

Nitrates

Experiment 140: Place some crystals of di-phenyl-amine in a wide-mouthed bottle, dissolve in concentrated sulphuric acid, and keep for use. Place about three drops of this di-phenyl-amine solution on a clean dry porcelain

surface. The di-phenyl-amine should be perfectly clear and colorless at this stage. Then add two or three drops of the solution to be tested. If a blue coloration is imparted to the di-phenyl-amine, it is indication of a nitrate. Other oxidizing agents will also give this blue coloration to di-phenyl-amine; and so, in the presence of strong oxidizers as chlorates, free chlorine, bromine, and iodine, this test is not proof for a nitrate.

Experiment 141: *Brown-ring Tests.*—Dissolve a crystal of ferrous sulphate in a little water, add a few drops of nitrate, then incline the test-tube and pour down its side about 2 c. c. of strong H_2SO_4. A brown ring forming at the surface of separation between the H_2SO_4 and the ferrous sulphate solution is proof presence of a nitrate.

$$2KNO_3 + 4H_2SO_4 + 10FeSO_4 = K_2SO_4 + 3Fe_2(SO_4)_3 + 2(FeSO_4)_2NO + 4H_2O.$$

This *brown-ring* test is exceedingly delicate. The ring is decomposed by heating, and is otherwise unstable.

Experiment 142 (*by Reduction to Ammonia*): Place a strip of metallic aluminium in a test-tube and cover with a solution of KOH. Fit the test-tube with a 1-hole stopper and delivery. Warm gently until hydrogen is given off slowly to expel any possible nitrogen compounds; cool, then add the solution to be tested, warm, and pass the gas through Nessler's solution. If a brown precipitate forms, it is proof of ammonia, and in the absence of ammonia in the original solution proves that a nitrate has been reduced. Nitrites, however, will give the same test.

Sulphates

Experiment 143: Remove heavy metals first, by warming with a solution of Na_2CO_3, and filtering; filtrate will contain Na_2So_4. Add $BaCl_2$. Result, $Na_2So_4 + BaCl_2 = ?$ Filter and wash. Transfer some of the precipitate to a

test-tube and add HCl and HNO_3. Warm. If the precipitate fails to dissolve, it is proof of $BaSO_4$, and conclusive proof that a sulphate was present.

Oxalates and Phosphates

Experiment 144: Remove heavy bases with Na_2CO_3 solution, as in the preceding test. Filter, after adding Na_2CO_3 to the solution to be tested, acidify slightly with HCl, boil to expel CO_2, and add $BaCl_2$. Ppt. is $BaSO_4$. Filter, and to filtrate add $CaCl_2$ and NH_4OH until alkaline. Precipitate is CaC_2O_4 and $Ca_3(PO_4)_2$. Filter, and treat the ppt. with acetic acid.

Residue is CaC_2O_4. Dissolve in dilute HNO_3, and add $KMnO_4$; pass gas into lime-water. A ppt. is $CaCO_3$; proof that an oxalate was present.

Solution H_3PO_4, $Ca(H_2PO_4)_2$, etc., add ammonium molybdate. A yellow ppt. is proof of a phosphate.

NOTE.—Arsenic acid gives a similar result, and, if present, must be removed by H_2S before applying this test.

Iodides and Bromides

Iodides

Experiment 145: The iodine ion of iodides is oxidized to free iodine by chlorine water. To the solution of an iodide (KI) add a little Cl water, then shake up with CS_2. A violet color shows free iodine. If too much Cl is used the I is oxidized to KIO_3, colorless.

$$2KI + Cl_2 = 2KCl + I_2.$$

Bromides

Experiment 146: To a solution of KBr add chlorine water, then shake up with CS_2. A brown or yellow globule is proof of free bromine. $KBr + Cl = KCl + Br$.

To Detect a Bromide in Presence of an Iodide

Experiment 147: In the presence of both an iodide and bromide an oxidizing agent will liberate iodine first, and its color will obscure that of the bromide. The iodine may be oxidized to colorless HIO_3 by chlorine water added drop by drop until the bromine color (brown) appears, or better, add slowly a saturated solution of $KClO_3$, then dilute H_2SO_4, warm gently, pour off a little of the solution after adding the $KClO_3$, and dilute H_2SO_4 and test with CS_2 to ascertain whether the iodine has all disappeared. As soon as the iodine is all oxidized to colorless HIO_3, the free bromine will appear, imparting the brown color to CS_2.

TESTS FOR SALTS

Method of Analysis for Salts Soluble in Water

Place about 1 gram of the substance in a test-tube and dissolve in 8 to 10 c. c. of water.

Flame Test

Experiment 148: Burn off a platinum wire in the flame until it ceases to impart any color to the flame, then dip in the solution and bring it immediately to the flame. View the flame through two thicknesses of blue glass. A reddish-violet color not obscured by the blue glass is proof of potassium. A luminous yellow imparted to the flame, and obscured by two thicknesses of blue glass, is proof of sodium.

Test for Ammonium

Experiment 149: Place about half an inch in depth of the solution in a test-tube, add 1 c. c. KOH, and warm. The odor of ammonia, or action on red litmus paper of the vapor, is proof for presence of ammonia.

For a more delicate test, fit the test-tube with a single-bored stopper and delivery-tube, and after adding KOH, pass the gas through 5 c. c. of Nessler's reagent. A brown ppt. is proof of ammonia.

Test for the Heavy Metals

Experiment 150: To about 5 c. c. of the solution of the original material add a few drops of HCl, and if a ppt. forms, continue adding until precipitation is according to the table for Group II.

Test for Acid Ions

Experiment 151: Dissolve a fresh portion of the original material; divide into the number of parts for which acid-ion tests are to be made. Using one part for each test, proved according to the outline for acid-ion tests.

Method of Analysis for Salts Insoluble in Water

Experiment 152: *a.* Take about a gram of the substance, place in a test-tube with water, and, by boiling a moment or two, bring into solution as much as will dissolve in water. Allow to settle, and filter. Test the portion that does dissolve for K, Na, NH_3, for the heavy metals, and for acid ions exactly as directed under analysis of salts soluble in water.

b. The portion which fails to dissolve in water is thoroughly washed on the filter-paper; it is then transferred to a test-tube, and warmed with dilute (1 to 1) HCl until as much as will dissolve has passed into solution. If a residue still remains, filter, evaporate the filtrate nearly to dryness to expel excess of HCl, dissolve in water, and analyze exactly as for water-solutions.

c. If the portion failing to dissolve in water does not

completely dissolve in HCl, take a fresh sample of that insoluble in water and dissolve as much of it as possible in dilute HNO_3. Test this as in preceding cases.

For substances failing to dissolve in HCl or HNO_3, consult Prescott and Johnson, Qualitative Analysis for *special methods.*

APPENDIX

Significance of Ions

"Suppose we have to deal with 50 bases and 50 acidic units of some kind, which may possibly unite to form 2,500 different compounds with distinct and individual properties. If the analyst were compelled to recognize these compounds singly, in the solid condition, he obviously would have to be familiar with the properties of each individual among the number; and were he to attempt to identify the individuals which might be present in a mixture, the task would be beyond accomplishment. Were the compounds not dissociated in solution, the problem would still be scarcely less difficult; but, being dissociable, his task becomes comparatively light. Since the properties of the solution of an ionized compound are merely the sum of the properties of its ions, and since the total number of ions with which we have assumed it necessary to deal is 100, it follows that the knowledge of 100 sets of properties is sufficient to identify the 2,500 compounds."

Electrolysis consists of separating the ionized bodies, held in solution, by means of an electric current. Liquids, unlike metals, are affected by the passage of an electric current through them, being decomposed with separation at the electrodes. The electrodes are the points where the current enters and leaves the liquid. Faraday suggested that the liquid which conducts the current had in solution a compound whose molecules are divided into freely moving particles, some of which were charged with positive

and the rest with negative electricity. Such compounds he called *electrolytes,* and to the broken parts of the molecules he gave the name *ions.* The positively charged he called *cathions,* and the negatively charged *anions.*

The attraction or neutralizing effect which ions of opposite polarities were supposed to exercise upon each other was thought to maintain the identity of the substance in solution until the electric current was passed; then the introduction of the electrodes of opposite polarities upset the equilibrium and caused the ions to migrate —the negative ions going toward the positive pole or electrode, and the positive ions going toward the negative electrode.

It has been proved by experiment that liquids that exhibit normal osmotic pressures and normal freezing-point depressions are non-conductors of electricity, and that their solutions are not electrolytes. But, on the other hand, the solutions which give abnormal osmotic pressures and freezing-point depressions are conductors of electricity and do contain ionized substances in solution, and the degree of conductivity is proportional to the amount of dissociation as measured by osmotic pressures; e. g., the two extremes would be represented by sugar with no conductivity and dissociation on the one hand, and by strong acids, bases, and salts with high conductivity and dissociation on the other. Hence, the difference in behavior of molecules held in solution is mainly due to their tendency of dissociation into ions; and the character of such solutions is distinguished by the reactions of the ions rather than by the properties of the undissociated molecules. In the case of sodium chloride, for example, the solution presents certain definite properties which are characteristic of chlorine and sodium ions, and not characteristic of the salt itself. While with sugar-solutions, such properties as are manifested are those of

sugar molecules alone, and no indication is to be seen in them of the nature of the constituent elements of sugar.

1. It may well then be asked, How does solution aid in chemical activity? By dissociation of the solute into ions, making it possible for them to combine with other ions.

2. How does heat aid chemical activity? By producing rapid vibrations of the molecules, which are thus more energetically broken into ions.

3. How may dilution aid chemical activity? By expanding the volume, thus decreasing the pressure and increasing the degree of dissociation.

Summary of a Few Facts of Dissociation

1. In any solution of acid, base, or salt, ions are partly free and partly united into electrically neutral molecules. The case is similar to the case of salt in contact with its own saturated solution, some salt dissolving and some salt recrystallizing. The above process takes place in producing equilibrium.

Another illustration is water in contact with its own vapor; some vapor is condensing and some water is evaporating. In the solution these two actions are going on: NaCl molecules are dissociated into Na and Cl; next, Na ions and Cl ions are uniting into NaCl molecules.

2. Salts of the simplest formula show great similarity and great uniformity in the degree of dissociation. (All are dissociated to the same extent.)

3. Acids show very different extents of dissociation.

First class, HCl, HNO_3, and H_2SO_4. Solutions of average strength of these acids dissociate to about 50 %.

Second class, H_3PO_4, H_2SO_3, and $HC_2H_3O_2$ dissociate to about 10 % in solutions of average strength.

Third class, H_2S, HCn, and H_2CO_3 dissociate to less than 1 %.

4. Bases are divided into two classes:

First class, the alkalis NaOH and KOH are soluble, and dissociate to about 50 %.

Second class, other bases are almost insoluble, but the parts that do dissolve are probably almost entirely dissociated.

5. The free ions never leave the solution, but as the solution is vaporized the ions collide more frequently, forming neutral molecules, so that molecules always pass through the undissociated state before the salt appears in solid form.

6. All undissociated molecules are chemically inactive; that means that chemical action always takes place between free ions.

7. Study of the third condition for end reaction.

We have already referred to the third condition, but it was necessary to study the behavior of acids, bases, and salts in water-solution before we could reach any definite conclusions.

We now return to our study of reactions, and see what is that third way for a substance to leave the field of action. Under the preceding topic we noticed that chemical action always took place between free ions, and that undissociated molecules are inactive. If this is the case, we can see that whenever undissociated molecules are formed they are removed from the field of action just as though the product were a gas or insoluble in water. Water is an example of such a substance. Hence, in simple reactions where water is a product, the reaction will complete itself. Two examples of this case. Acid with an oxide or hydroxide ($NaOH + HCl$). The ions are Na, OH, H, and Cl. The OH and the H unite and form undissociated water; thus between a base and an acid, the reaction will always be practically complete.

The reaction between an acid and the salt of another

acid will be more or less complete, according as one acid is more or less dissociated than the other. $HCl + Na(C_2H_3O_2)$. The ions are Na, Cl, H, and $C_2H_3O_2$.

Under topic 3 we have seen that $HC_2H_3O_2$ is slightly dissociated; hence, when H_2 ions join $C_2H_3O_2$ ions a weakly dissociated set of $H(C_2H_3O_2)$ molecules result. The acid is practically removed from the field of action and the reaction will nearly complete itself. $NaC_2H_3O_2 + HCl = NaCl + (HC_2H_3O_2)$.

8. Summary of conditions for end-reactions.

a. Reaction between base and acid is practically complete because undissociated water is a product.

b. Between salt of an acid and another acid:

1. The reaction is practically complete if one of the acids is strongly dissociated while the other is slightly dissociated.
2. The reaction is complete if, on account of comparatively high temperature, one acid becomes a gas.

c. Reactions between salts are complete if one of the salts formed is insoluble.

9. How to predict reactions. (From Torrey.)

From the foregoing discussion it will be seen that, in order to predict what will happen when two salts, an acid and a salt, or a base and a salt, come together in water-solution, we need to know the following: *Whether the union of any two ions present in the solution will result in a substance which is insoluble, volatile, or weakly dissociated.* We have already named (2, 3, 4, 5, under facts of dissociation) those substances which are strongly or weakly dissociated. We now give a table of solubilities, and tabulate some materials, insoluble or slightly soluble in water.

Principles of Precipitation

1. All metallic oxides and hydroxides are insoluble, except those of the alkali metals; those of Ba, Sr, and Ca are slightly soluble. Fixed alkali hydroxides precipitate the common metallic salts (not As^{III}). The precipitate is a hydroxide, except that Ag^{I}, Sb^{III}, Hg^{I}, and Hg^{II} form the oxides, and stannic compounds form stannic acid, H_2SnO_3. The precipitate redissolves in excess of the reagent in eight cases: Pb, Sn^{II}, Sn^{IV}, Sb^{III}, Sb^{V}, Cr, Al, and Zn. $Cr(OH)_3$ is reprecipitated on boiling the alkaline solution.

2. All nitrates, chlorates, and acetates are soluble; but salts of Cu^{I}, Bi, Sn, Sb, and the oxysalts of mercury require some free acid to hold them in solution.

3. Fixed alkali carbonates precipitate (not As^{III}) all other metallic salts. The precipitate is an oxide with Sb^{III}, an hydroxide with SN^{II}, Al^{III}, Cr^{III}, and Fe^{III}; with antimonic compounds it is antimonic acid, H_3SbO_4; with stannic compounds, stannic acid, H_2SnO_3; and in other cases a normal or basis carbonate, except for $HgCl_2$, which forms an oxychloride.

4. Ammonium hydroxide precipitates the first four groups (not As^{III}). Fe^{II}, Mn^{II}, and Mg^{II} are precipitated but partially, or not at all, in the presence of ammonium salts. The precipitate is a hydroxide except with Ag^{I} and Sb^{III}, which form the oxides; with Hg^{II}, which forms mercuric ammonium compounds; with Pb^{II}, which forms a basis salt; and with Hg^{I}, which forms mercuric ammonium compounds and free Hg. The precipitate redissolves in excess of the reagent in six cases: Ag, Cu, Cd, Co, Ni, and Zn.

5. Hydrochloric acid or a chloride precipitates Ag^{I}, Hg^{I}, and Pb^{II}, as the chlorides, Pb^{II} incompletely.

6. Hydrobromic acid and bromides precipitate the bromides in the same cases.

7. Hydriodic acid or an iodide precipitates Ag^{I}, Hg^{I}, Pb^{II}, Hg^{II}, and Cu^{I} as the iodides; Cu^{II} as Cu^{I} with liberation of iodine. Fe^{III} becomes Fe^{II}, and H_3AsO_4 becomes H_3AsO_3, with liberation of iodine.

8. The sulphides of the first four groups are insoluble. H_2S precipitates in the presence of dilute acid the first two groups as sulphides. It reduces Fe^{III} to Fe^{II}.

9. Soluble sulphides, or H_2S in alkaline solution, precipitate the first four groups as sulphides, except Al^{III} and Cr^{III}, which are precipitated as hydroxides, and Fe^{III}, which forms FeS and S.

10. Sulphuric acid, a sulphate, precipitates Pb^{II}, Ba^{II}, Sr^{II}, Hg^{I}, Ca^{II}, Hg^{I}, and Ca^{II} incompletely.

Qualitative Analysis of Metals

SUMMARY OF EXPERIMENTS WITH Pb, Ag, Hg^{I}

(GROUP I)

Table for the Analysis of the Silver Group

The precipitate is washed on the filter with one or two small portions of cold water; then treated with hot water, and filtered.

Residue: AgCl, HgCl. Digest with ammonium hydroxide and filter.	Solution: $PbCl_2$. Divide the solution into four portions and test with the following: (1) H_2SO_4, giving $PbSO_4$, white. (2) H_2S, giving PbS, black. (3) K_2CrO_4, giving $PbCrO_4$, yellow. (4) KI, giving PbI_2 yellow.
Residue: Hg and NH_2HgCl, black. (Oxychloride, white.) The black color is the evidence of mercury, and of the presence of Hg in the mercurous condition.	Solution: $(NH_3)AgCl_2$). Boil, to expel the excess of ammonium hydroxide, and acidulate with nitric acid. A precipitate is AgCl.

SUMMARY OF EXPERIMENTS WITH As, Sb, Sn

(GROUP II A)

Analysis of the Metals of the Tin Group

Pass H_2S through the filtrate from Group I as long as a precipitate continues to form. Filter, and save the filtrate for Group III. The precipitate consists of the sulphides of As, Sb, Sn, Hg, Pb, Bi, Cu, Cd. Pierce the point of the filter, and rinse the ppt. into a test-tube with a small amount of yellow ammonium sulphide. Digest for a few minutes without warming, then filter, and save the residue for Division B of Group II. The solution from yellow ammonium sulphide contains the sulphide of As, Sb, and Sn (Group II A). Precipitate this solution by adding dilute HCl. The precipitate is As_2S_3, As_2S_5, Sb_2S_5, SnS_2. Filter, and digest the ppt. with solution of $(NH_4)_2CO_3$. Filter.

Residue undissolved in $(NH_4)_2CO_3$ is SnS_2, Sb_2S_5. Orange yellow, if Sb; brown, if containing Sn. Dissolve in nitrohydrochloric acid to form pyroantimonic-acid solution, then add $(NH_4)_2CO_3$, precipitating metastannic acid, white, insoluble in fixed-alkali carbonates, distinction from antimony. The presence of Sb and Sn may be further verified by the Marsh test. (See text.) Also see TIN for short test for tin.	Solution: Contains arsenates and sulparsenates. Add HCl. A yellow ppt. is As_2S_3 and As_2S_5, indication of arsenic. The presence of arsenic may be further verified by dissolving the As_2S_5 in a little free-chlorine solution formed by adding HCl to a crystal of $KClO_3$, then applying in the Marsh-test for arsenic.

SUMMARY OF EXPERIMENTS WITH Hg^{II}, Cu, Cd, Bi

(GROUP II B)

Analysis of the Metals of the Copper Group

Sulphides insoluble in yellow ammonium sulphide, HgS, PbS, Bi_2S_3, CuS, CdS, S.

The well-washed precipitate is boiled with dilute (2N)HNO_3.

<table>
<tr><td rowspan="6">Residue: HgS (black): S Dissolve in nitrohydrochloric acid and boil to expel free Cl solution. $HgCl_2$. Add $SnCl_2$, precipitating HgCl, white, or Hg, gray.</td><td colspan="5">Filtrate: Nitrates of Pb, Cu, Cd, Bi. Evaporate almost dry in the porcelain dish to free from HNO_3, digest with hot water, and filter.</td></tr>
<tr><td rowspan="5">Residue: Basic Bi salts. Wash, and add to the precipitate on the paper hot Na_2SnO_2 solution, producing black metallic Bi.</td><td colspan="4">Filtrate: Nitrates of Pb, Cu, Cd, (Bi). Add dilute H_2SO_4, shake, and allow to stand.</td></tr>
<tr><td rowspan="4">Precipitate: $PbSO_4$, white, soluble in NaOH.</td><td colspan="3">Filtrate: Nitrates of Cu, Cd, (Bi). Add NH_4OH.</td></tr>
<tr><td rowspan="3">Precipitate: $Bi(OH)_3$, white. Wash, and test with stannite, as above.</td><td rowspan="3">A blue color in the filtrate is evidence of Cu. Decolorize with KCN and pass in H_2S; a yellow to green precipitate is CdS.</td><td colspan="2">If the filtrate is colorless, divide into two portions.</td></tr>
<tr><td>In one portion test for traces of Cu by concentrating by evaporation, acidifying with acetic acid, and adding $K_4Fe(CN)_6$. A brown precipitate is $Cu_2Fe(CN)_6$.</td><td>Into the other portion pass H_2S. A yellow to green precipitate is CdS.</td></tr>
</table>

SUMMARY OF EXPERIMENTS WITH Fe, Al, Cr

(GROUP III)

Analysis of the Iron Group (in Absence of Phosphate, etc.)

Boil the filtrate from Group II in an open dish to expel H_2S, add a few drops of HNO_3, and boil to oxidize ferrous iron. Immediately add NH_4Cl and excess NH_4OH; if a precipitate occurs, filter, and wash, reserving the filtrate for the Zinc Group.

Precipitate: $Fe(OH)_3$, brown; $Cr(OH)_3$, green; $Al(OH)_3$, white.

Dissolve a portion in HNO_3, add $KClO_3$ crystals, and boil to small bulk. Dilute, and add NH_4OH in a slight excess. Filter, and wash.

Precipitate: $Fe(OH)_3$. Dissolve in HCl and add KCNS. A blood-red solution, $Fe(CNS)_3$. Test the original for Fe^{II} with $K_3Fe(CN)_6$; for Fe^{III} with KCNS.

Filtrate: $(NH_4)_2CrO_4$. Acidify with acetic acid and add lead acetate. A yellow precipitate is $PbCrO_4$ .002 mg. delicacy.

Cr may be detected in this way.

$Cr(OH)_3$ may also be oxidized to chromate by fusion in the porcelain crucible with KNO_3 or $KClO_3$.

Boil another portion with very dilute NaOH solution. Filter. Filtrate: $NaAlO_2$. Acidify slightly with HCl and add $(NH_4)_2CO_3$. Or add to the alkaline solution NH_4Cl in excess and boil. A white precipitate is $Al(OH)_3$.

SUMMARY OF EXPERIMENTS WITH C. Ni, Mn, Zn

(GROUP IV)

Analysis of the Zinc Group

Into the ammoniacal solution from Group III pass H_2S; if a precipitate appears, warm, filter, and wash, preserving the filtrate for the following group. Precipitate: CoS (black), NiS (black), MnS (flesh-colored), ZnS (white). Treat on the filter with cold dilute (N)HCl.

<table>
<tr><td colspan="2">Residue: CoS, NiS.</td><td colspan="2" rowspan="2">Solution:
$ZnCl_2$, $MnCl_2$, H_2S.</td></tr>
<tr><td>For Co, test in the borax bead. If Ni is in large excess, dissolve the sulphides in HCl, with a drop of HNO_3, precipitate Co with nitroso-B-naphthol, and test this precipitate on the bead; or dissolve the sulphides in HNO_3 and treat with $NaHCO_3$ and H_2O_2.</td><td>For Ni, dissolve in HNO_3, evaporate excess of acid, treat with Bromine water and NaOH, and boil, precipitating $Ni(OH)_2$, black. Wash the precipitate very thoroughly, and add to it on the paper hot dilute KI solution; acidify the filtrate. If Ni is present, I° will be liberated, yellow in water, violet in CS_2 solution.</td><td colspan="2">Boil to expel H_2S. Cool, and add NaOH in excess. Filter.</td></tr>
<tr><td colspan="2"></td><td>Precipitate: $Mn(OH_2)$; boil with Pb_3O_4 and HNO_3; let settle. A violet solution, $HMnO_4$.</td><td>Solution: Na_2ZnO_2. Add H_2S. A grayish-white precipitate, ZnS.</td></tr>
</table>

SUMMARY OF EXPERIMENTS WITH Ba, Sr, Ca, Mg

(GROUP V)

Analysis of the Calcium Group

To the filtrate from the fourth group add ammonium hydroxide and carbonate; warm, but do not boil. Filter, and wash.

Precipitate: $BaCO_3$, $SrCO_3$, $CaCO_3$.

Dissolve the well-washed precipitate in dilute acetic acid.

Solution: $Ba(C_2H_3O_2)_2$, $Sr(C_2H_3O_2)_2$, $Ca(C_2H_3O_2)_2$.

To a small portion of the solution add potassium dichromate; if a precipitate appears, add the reagent to the whole solution as long as a precipitate is produced, and filter.

<table>
<tr>
<td>Precipitate: $BaCrO_4$, yellow. The precipitate is soluble in hydrochloric acid, and this solution is precipitated by sulphuric acid, as $BaSO_4$, insoluble in acids. Test the above hydrochloric acid solution with a loop of platinum wire in the flame.</td>
<td colspan="2">Solution: $Sr(C_2H_3O_2)_2$, $Ca(C_2H_3O_2)_2$, $(K_2Cr_2O_7)$.
Reprecipitate with ammonium hydroxide and carbonate; filter, wash the precipitate, and dissolve it by acetic acid.
Solution: $Sr(C_2H_3O_2)_2$, $Ca(C_2H_3O_2)_2$.
Divide into two portions.</td>
</tr>
<tr>
<td></td>
<td>For Strontium:
To a portion add solution of calcium sulphate; boil, and leave for about ten minutes. A precipitate indicates strontium, $SrSO_4$. Test another portion of the solution on a loop of platinum wire by the flame.</td>
<td>For Calcium:
Add a solution of potassium sulphate, boil, allow to stand ten minutes, and filter to insure the absence of strontium. To the filtrate (or solution not precipitated) add ammonium oxalate.
A precipitate (CaC_2O_4), white, insoluble in acetic acid, soluble in hydrochloric, indicates calcium. Test by flame.</td>
</tr>
</table>

Solution: Salts of Magnesium and the Alkali Metals. To the solution in which $(NH_4)_2CO_3$ fails to give a

precipitate, add a drop or two of $(NH_4)_2SO_4$, and then a few drops of $(NH_4)C_2O_4$. If a precipitate is obtained (showing presence of traces of Ba, Sr, or Ca), filter, and to a portion of the clear filtrate add Na_2HPO_4. A white precipitate ($MgNH_4PO_4$) indicates magnesium.

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